Docket No.: **1152-014A**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	)
Bradley L. Grunden et al.	) Group Art Unit: 1771
Serial No.: 10/676,901	) Examiner: Matzek, Matthew D.
Filed: October 1, 2003	)

For: ELECTROSTATIC CHARGE DISSIPATING HARD LAMINATE SURFACES

DECLARATION UNDER 37 CFR 1.131

Kenneth J. Heater declares and states as follows:

1. He is a named inventor in the above-identified application and a representative of the assignee of the above-identified application, METSS Corporation, and makes this declaration to provide evidence of the completion of the invention described and recited in the above-identified application in the United States of America prior to December 1, 1999.

2. He has read and is familiar with the Office Action dated May 20, 2005, in the above-identified application in which Yeager et al., United States Publication No. 2001/0053820, has been relied upon to reject Claims 1-5, 13, 15, 16, and 19 under 35 U.S.C. 103(a).

3. He has been informed and believes that the earliest effective date of said United States Publication No. 2001/0053820 is December 1, 1999.

4. Prior to December 1, 1999, the invention described in the above-identified application was completed in the United States of America. Provisional application Serial No. 60/415,833 was filed on October 3, 2002, less than one year after United States Publication No. 2001/0053820 was published.

5. Prior to December 1, 1999, under my direction, METSS Corp. submitted to the U.S. Department of Defense a Small Business Innovation Research (SBIR) Program

Proposal Cover Sheet and Project Summary for a Phase II program describing its proposal for developing static dissipative hard laminate surfaces that had been prepared by METSS Corp. The proposal included a description of the use of nanophase materials, polymer blend technology, and polymer synthesis techniques to create improved technologies for hard laminate worksurfaces. The selection of appropriate materials and technology required to develop antistatic characteristics on the melamine surface was also noted in its proposal. It was already known to blend melamine resins with intrinsic semiconducting polymers. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit A is a copy of a U.S. Department of Defense Small Business Innovation Research (SBIR) Program Proposal Cover Sheet and Project Summary.

6. As further evidence in support of the averments in paragraph 4, above, attached hereto as Exhibit B is a copy of a memorandum from the Department of the Air Force to the METSS Corporation notifying METSS of the acceptance of its proposal, as discussed in paragraph 5, above, which was received prior to December 1, 1999.

7. Prior to December 1, 1999, at my direction, Julius Brodbeck, an employee of the U.S. Air Force, Air Force Research Laboratory (AFRL), mounted three laminate samples on ¾" furniture grade plywood. Samples 1 and 2 were glued to the plywood and Sample 14 was attached using eight bolts around the edges to hold it flat. Sample 14 was evaluated by Julius Brodbeck for Rtt (top to top resistance), uniformity, and static dissipation at 10% and 50% relative humidity and found to be satisfactory. To test for uniformity of resistance, a 2.5" diameter probe consisting of an outside ring (guard, negative) and a positively charged disk about 1" in diameter at the center of the ring were used. When the Resistivity was tested at various points on the sample, the values at the different points were consistent with each other, which is a good indication of surface uniformity. To test for charge dissipation, a circular aluminum disk was charged with either +1000 or –1000 volts. The disk was then brought into contact with the sample for five seconds, and a reading of the disk was taken again. Acceptable values are between +200 and –200 volts. Sample 14 fell within these values on four separate trials. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit C is a copy of a memo from Julius Brodbeck to me, Mike

Manders (USAF/AFRL), Steve Gerken (USAF/AFRL), and Mick Hitchcock (USAF/AFRL) analyzing METSS Corp. Samples 1, 2, & 14 for Top to Top Resistance and Static Dissipation at both 10% and 50% Relative Humidity (RH) and 72°F, which was written prior to December 1, 1999.

8. Prior to December 1, 1999, a product was made which consisted of solution concentrations of a commercially available intrinsically conductive polymer (Bayer Baytron® P) of up to 10% blended with a standard melamine formaldehyde resin used in the production of commercial laminates. The formulations were stable and 100% water soluble. The product was tested according to the procedure of paragraphs 5 and 6 of Exhibit H, and the tests showed that an inherently conducting polymer (ICP) content of less than 1% would be sufficient to address the ESD requirements set forth by the Air Force. I contemplated replacing the current curing agent ("Part B") used by Formica to cure their melamine resins with a new "Part B" that contained the ICP additive. The METSS ICP-B is an aqueous solution that mixes readily with the melamine resin system and has the right pH to effect cure in the same manner as the existing Part B. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit D is a copy of an Invention Disclosure summarizing the invention, which was written by me prior to December 1, 1999.

9. Prior to December 1, 1999, a method of addressing the ESD issue through use of nanophase additives, polymer blending, and the use or synthesis of intrinsically conducting polymers or co-polymers was proposed. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit E is a copy of a monthly status report which was written by me prior to December 1, 1999.

10. Prior to December 1, 1999, initial experiments were conducted using nanoparticles in a model resin system (polyvinyl alcohol) to assess the general dispersion characteristics and the effects of loading levels on resin properties. Particle size analysis measurements were performed using a CAPA 500 Centrifugal Automatic Particle Analyzer. Isoelectric point measurements were performed using a MATEC ESA 8000. The following nanophase additives were used in the ESD formulation development efforts: $SbSnO_2$, Printex L6, Printex L, and carbon black nanotubes. Under my direction, METSS staff carried out a series of tests on sonicated solutions of $SbSnO_2$ in

water in order to determine optimum conditions for particle deagglomeration. It was determined that, from a practical standpoint, it would not be difficult to disperse the $SbSnO_2$ nanoparticles in the melamine resin system at any practical pH level. Some initial ESD test results (as performed by the Air Force program monitor) on the Baytron sample demonstrated proper conductivity and passed the ESD test at 37% relative humidity. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit F is a copy of a monthly status report which was written by me prior to December 1, 1999.

11. Prior to December 1, 1999, the following nanophase additives were used in our ESD formulation development efforts: $SbSnO_2$, Printex L6, Printex L, carbon black nanotubes, fluorine doped ZnO, and other nanophase carbon. Under my direction, METSS staff also carried out a series of particle deagglomeration experiments on a carbon black sample at pH = 2, using a sonic bath and sonic horn. By incorporating polyvinyl alcohol into the aqueous suspension, it was possible to obtain the desired dispersion. A ZnO Dispersion Stabilization Study was also conducted, and it was found that, by using 1-2 wt. % Acrysol WS-32 as a stabilizer, stable dispersion was obtained at low pH when 5 wt. % Duramax D-3021 was added. Uniformly dispersed carbon fibers in water or isopropyl alcohol were obtained by using sonication in a sonic bath. Additionally, a series of laminates was prepared from substrate materials and cellulose papers impregnated with solutions of varying ratios of Baytron® P to melamine formaldehyde resin. Lastly, compliance was achieved with the Air Force requirement for less than 200V of remaining charge after 5 seconds of dissipation from 1000V at all humidity levels tested at loading levels down to the 1% tested. As evidence in support of the averments in this paragraph and in paragraph 4, above, attached hereto as Exhibit G is a copy of a monthly status report which was written by me prior to December 1, 1999.

12. As still further evidence in support of the averments in paragraphs 4 and 11, above, attached hereto as Exhibit H is a copy of a memo written by me to Julius Brodbeck, Mike Manders, Steve Gerken, and Michael Hitchcock (USAF/AFRL) detailing the Phase I Technical Feasibility Demonstration, which was written prior to December 1, 1999.

13. Dates have been masked out of the attached exhibits and all of the actual dates that have been masked out are prior to December 1, 1999.

He further declares that all statements made herein of his own knowledge are true and that all statements made of information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

9/11/05
Date

Kenneth J. Heater

APPENDIX A

U.S. DEPARTMENT OF DEFENSE

SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM
PROPOSAL COVER SHEET

Failure to fill in all appropriate
spaces may cause your proposal to be disqualified

TOPIC NUMBER: AF99-146

PROPOSAL TITLE: Develpment of Static Dissipative Hard Laminates Surfaces

FIRM NAME: METSS Corp.

MAIL ADDRESS: 720 G Lakeview Plaza Blvd.

CITY: Columbus STATE: OH ZIP: 43085

PROPOSED COST: $99,866 PHASE I OR II: I PROPOSAL PROPOSED DURATION: 9MO IN MONTHS

BUSINESS CERTIFICATION:

	YES	NO
▸ Are you a small business as described in paragraph 2.2?	X	☐
▸ Number of employees including all affiliates (average for preceding 12 months): 14		
▸ Are you a socially and economically disadvantaged business as defined in paragraph 2.3? (Collected for statistical purposes only)	☐	X
▸ Are you a woman-owned small business as described in paragraph 2.4? (Collected for statistical purposes only)	☐	X
▸ Have you submitted proposals or received awards containing a significant amount of essentially equivalent work under other DoD or federal program solicitations? If yes, list the name(s) of the agency or DoD component, submission date, and Topic Number in the spaces below.	☐	X

PROJECT MANAGER/PRINCIPAL INVESTIGATOR

NAME: Kenneth J. Heater, Ph.D.

TITLE: Senior Scientist

PH: 614-842-6600 FAX: 614-842-6601

CORPORATE OFFICIAL (BUSINESS)

NAME: Kenneth J. Heater, Ph.D.

TITLE: Vice President

PH: 614-842-6600 FAX: 614-842-6601

For any purpose other than to evaluate the proposal, this data except Appendix A and B shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.

PROPRIETARY INFORMATION:

Before signing below, please read the cautionary note at Section 3.7

K Heater
SIGNATURE OF PRINCIPAL INVESTIGATOR DATE

K Heater
SIGNATURE OF CORPORATE BUSINESS OFFICIAL DATE

U.S. DEPARTMENT OF DEFENSE

SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM

PROJECT SUMMARY

Failure to fill in all appropriate spaces may cause your proposal to be disqualified

TOPIC NUMBER: AF99-146

PROPOSAL TITLE: Development of Static Dissipative Hard Laminate Surfaces

FIRM NAME: METSS Corporation

PHASE I or II PROPOSAL: I

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information/data.)

Current materials used for static discharge applications in hard worksurface laminates do not provide the required levels of charge dissipation under all conditions. The need exists for an integrated conductive laminate system that enables static charge to be safely and effectively dissipated from equipment and components placed in contact with the worksurface, thereby affording ESD protection to equipment and personnel. Although current laminates posses satisfactory electrical resistivity, efficient charge dissipation has been hampered by the inability of the systems to overcome the small contact area between equipment and hard worksurfaces in conditions of low humidity. Under the proposed program, METSS will investigate incorporating recent advances in nanophase materials processing, polymer blend technology, and polymer synthesis techniques to create improved technologies for hard laminate worksurfaces. Nanophase additives and/or careful control of polymer blending conditions with compatibilizers offer the potential to maximize the amount of conductive phase in the resin yet minimize additive weight and volume, thereby providing homogeneous and stable ESD protection beyond that currently available without adversely effecting mechanical properties. METSS is uniquely qualified to develop the new laminate system, possessing the necessary expertise in polymers and polymer processing, testing and evaluation of composite materials and expertise and practical experience in developing advanced additives for resin systems and formulating resins with advanced properties.

Anticipated Benefits/Potential Commercial Applications of the Research or Development.

A hard, conductive laminate resin system incorporating nanophase materials, polymer blend technologies and/or formulation of intrinsically conducting polymer systems would impart precisely controlled levels of antistatic performance into a hard, structurally sound laminate without requiring specialized manufacturing techniques. The technology has significant commercial potential in areas including antistatic floor and benchtop applications, protective textiles and advanced coatings.

List a maximum of 8 Key Words or short (2-3 word) phrases that describe the Project.

Conductive	Blend
Antistatic	Copolymer
Nanophase	Resin
Additive	Laminate

Nothing on this page is classified or proprietary information/data

C. IDENTIFICATION AND SIGNIFICANCE OF THE PROBLEM OR OPPORTUNITY

C.1 Program Overview

Recent advances in materials processing techniques provide the opportunity to combine specialty nanophase particle technology with melamine oligomer systems to produce a conductive melamine-based resin system with precisely controlled antistatic properties for workspace applications. Alternatively, conductive laminate systems may be designed based on developments in core-shell polymer blend technology and/or novel polymerization techniques to create durable, conductive polymer matrices. Under the proposed program, METSS will demonstrate the technical feasibility of using these emerging technologies to develop a new conductive laminate system which exhibits carefully defined antistatic properties while still retaining requisite mechanical properties. Key property testing will be performed under the Phase I program to support and direct prototype design efforts. A preliminary prototype will be produced under the Phase I program. Further prototype design, testing, optimization, and qualification efforts will be performed under the Phase II program. Preliminary product commercialization opportunities will be identified throughout the course of the Phase I program as candidate systems are developed. More focused product commercialization efforts will be pursued through close interactions with program commercialization partners under the Phase II program, based on technologies identified throughout the Phase I program.

Having been involved in a number of materials technology development programs utilizing advanced concepts polymer/resin chemistry, METSS is intimately familiar with the development of new resins and additives for high performance resin systems. Based on this experience and other ongoing research efforts, METSS is confident that the probability of success of this program is high, offering excellent prospects for development into a mature technology.

C.2 Statement of Problem

The primary objective of the SBIR program is to develop a structurally sound laminate system with controlled electrical properties (conductivity), which is suitable for use in workstation applications as an integrated system for protection against ESD events. Currently, softer, more pliable worksurfaces perform satisfactorily, as the material is able to conform to any surface irregularities in contact with the worksurface, thereby maximizing contact area and providing sufficient pathways for the charge to dissipate to ground. However, while current hard laminate systems based on melamine resin oligomers exhibit acceptable resistivity and mechanical properties, these materials do not perform to expectations at low humidity, since the number of conductive pathways to ground through which static charge can be safely dissipated is reduced. This is because the actual interfacial area between the worksurface and component, across which safe discharge must take place, is significantly less than the apparent area. This is due to the presence of microscopic surface features such as peaks and ridges on the worksurface, which decrease the actual area of contact and reduce the pathways to ground by a significant amount. Compounding this is the fact that current conductive additives are typically too large to occupy positions immediately adjacent to the surface in these surface features and are readily removed or fluffed by sliding contact with equipment and components on the worksurface. Particle removal (dusting) and/or fluffing can render the surface features relatively insulative, thereby exacerbating the problem. It is only in higher humidity environments, where water vapor can fill the spaces between the surface features and provide a fluid conductive path across the interface to ground, that the worksurfaces perform satisfactorily.

The proposed program will address issues of ESD protection in melamine based resins, thereby providing a reliable and resilient laminate for long-term ESD protection. This will be accomplished by applying new advances in blending methods, polymer formulation expertise and processing techniques for specialized additive materials that directly address the problems stated above, providing new capabilities

for integrating conductive technology into melamine resin laminates. The approach proposed by METSS will minimize negative impact on laminate structural integrity and longevity.

C. 3 Design Elements

The key elements that must be addressed in order to achieve the stated objectives of this SBIR include:

Materials - the resin must be constructed of materials which:

- retain sufficient structural integrity of the worksurface (this will be dependent both on the structural properties of the materials themselves, and on the interfacial adhesion properties between the resin and additive materials, compatibility of blend phases, etc.)
- exhibit excellent static dissipative performance characteristics with conductivity in a precisely determined range
- exhibit physical flexibility and resilience (impact resistance, scratch resistance, longevity).

Design - the design of the laminate system should address the following elements:

- physical design requirements dictated by the application (size, thickness, etc.)
- structural design requirements dictated by the application (strength, durability, etc.)
- control of particle additive/conductive phase distribution for conductivity and structural integrity.
- design objectives must be met in the most cost-effective manner possible

Fabrication - laminate fabrication should be consistent with the following objectives:

- manufacturing should not require specialized techniques to achieve the required material properties
- resin material should be readily incorporated into current state-of-the-art practices
- the nature of the laminate design and manufacturing process should permit in-service repair where necessary.

METSS will address each of the relevant issues under the proposed SBIR program, reviewing and generating critical information to design a detailed prototype, incorporating hard scientific data to support property and performance assessments, and demonstrated manufacturing capability to support design efforts and illustrate product feasibility.

C. 4 Potential Commercial Opportunities

The technology developed under the Phase I program will provide the basis for highly focused Phase II refinements, further developments and application-specific modifications of the conductive resins and integrated laminate structures. The potential exists in several industries and applications to develop new markets and/or enhance existing practices. These include:

- Industrial and domestic worksurfaces – there are literally hundreds of applications where static charge buildup and ESD events impact on the work and home environment where the proposed technology would be of enormous benefit.

- Floor and wall coverings – industrial cleanrooms, domestic/household flooring materials, automobile floor mats. Technology could be incorporated into bulk material or into coating, topcoat etc. (e.g. floor lacquer)
- Hospital applications – floor, wall and fixture coatings where the presence of flammable gases and chemicals, (anaesthetic, alcohols etc.) makes it highly desirable to prevent electrostatic discharge
- Equipment storage – especially in low relative humidity environments, storage of delicate electrical componentry etc.
- Laboratory/industrial safety – safety sheets, mats and worksurfaces for use in hazardous, flammable environments, safety footwear, anti-static wands.
- Military and advanced commercial applications - the technologies developed under the program can be used to address other needs where advances in electrical properties of polymers could have a substantial impact, including advanced coatings for aircraft canopies, windscreens, monitors, etc.

During the Phase I program, METSS will work proactively to evaluate the potential of these and other opportunities, identify new commercialization possibilities and establish preferred commercialization routes to be aggressively pursued under the Phase II proposal. The stated SBIR application is closely aligned with many possible domestic and industrial applications, which should provide opportunities for discussions with a number of potential commercialization partners. In the past, METSS has been very successful at identifying and establishing solid working relationships with industrial partners. Such partners provide invaluable support to Phase II product scale-up efforts, as well as the manufacturing, marketing, sales, and distribution efforts required for product commercialization. The applicability of this technology across a broad range of industries should guarantee that industrial partners for the program are readily identified, and ensure that efforts to pursue commercialization will provide a smooth and timely transition of the technologies developed under this program from the laboratory to the field.

METSS will work directly with current manufacturers of ESD laminates to support product commercialization within this market segment. During the course of the Phase I program, METSS will pursue discussions with companies like NEVAMR, a division of International Paper and one of the primary suppliers of ESD laminates, to support efforts in this regard.

D. PHASE I TECHNICAL OBJECTIVES

Under the proposed Phase I SBIR program, METSS will clearly demonstrate the technical feasibility of developing a specialized conductive laminate system for workstation ESD control. The proposed program will include the use of nanophase materials as conductive additives, polymer blend technology, or formulating an intrinsically conductive polymer system to develop resin systems capable of addressing the program requirements. METSS will achieve this objective by accomplishing the following:

- Identification of critical design and performance elements of the laminate structure through technical meetings and discussions.
- Performance of a critical technology review to select most feasible combinations of matrix and additive materials, and establish well defined design objectives.
- Development of production methodologies, incorporating combinations of specialized additives, blend systems and/or copolymer series' that are directly compatible with the stated application.

- Development of test methodologies for establishment of compliance with military specifications and other design criteria.
- Fabrication of prototype system(s), which exhibit the best combination of structural integrity, conductivity and other required characteristics.

The ultimate goal of the Phase I SBIR program is to demonstrate the feasibility of producing a controlled conductivity laminate system through the selection and use of specific technologies, based on conductive nanophase additives, polymer blends and intrinsically conductive polymers/copolymers. METSS will initially concentrate on achieving the program through the use of nanophase additives, progressing to blend technologies and intrinsically conductive polymer/copolymer systems only if the technical objectives of the program are unable to be met by the nanophase additive approach.

METSS intends to use the data generated in the Phase I effort to support a detailed Phase II program agenda involving development, testing, and optimization efforts, to be presented in a Phase II proposal provided by METSS at the sponsor's request. Under the Phase II effort, METSS will fully develop and refine the concepts identified under Phase I, setting the foundation for further development of the technology and for focused product commercialization and distribution efforts immediately after the Phase II program. *The results of the Phase I program will be presented in a detailed report to the program sponsor that will include support documentation for Phase I materials selection and development efforts, along with detailed processing and property descriptions of a prototype ESD compliant laminate system.* Design and fabrication concepts developed under the Phase I program will be reviewed with the program sponsor during a final program review meeting.

E. PHASE I WORK PLAN

The Phase I program work plan is presented in this section. Each task is identified and discussed in detail. The qualified staff of METSS will perform all of the work performed under this program. Dr. Kenneth Heater will serve as principal investigator for the proposed effort. Additional technical support will be provided by senior staff engineers/scientists of METSS with specific expertise in polymeric materials processing, synthesis, fabrication, testing, and product evaluation. All work performed by METSS staff will be conducted at METSS' laboratory facility located in Columbus, OH. The close proximity of METSS' facility to WPAFB will greatly enhance the program efforts through ease of interaction between METSS personnel and the program sponsor.

A nine (9) month period of performance has been scheduled for the Phase I program. However, the technical feasibility of the proposed concept will be demonstrated during the first six (6) months of the program to accelerate the transition to the Phase II program efforts. A program schedule is presented in Table 1. The Phase I program tasks can be completed within the confines of the proposed budget, within the proposed time schedule.

METSS will adopt a tiered approach to the program, addressing each tier in order of increasing complexity. In this manner, METSS will essentially investigate the simplest approach to the problem first, proceeding to the more complex approaches only if results indicate that the likelihood of success of earlier approaches is small.

Task 1. Start of Work Meeting

A start of work meeting should be held between the program sponsor and METSS to include discussion of the following important program aspects:

Table 1: Phase I Work Schedule

Task Description	Month								
	1	2	3	4	5	6	7	8	9
Task 1. Start of Work Meeting									
Task 2. Technical Review									
Task 3. Processing & Fabrication Development									
Task 4. Testing and Evaluation									
Task 5. Prototype Fabrication and Testing									
Task 6. Administrative & Reporting		♦	♦	♦	♦	♦	♦	♦	F

F = final report

1. Product Requirements: METSS would like to conduct an informal review of critical design elements for the resin laminate system. A brief description by the program sponsor on the service conditions and expected functionality of the laminates would be of great benefit to the program, giving METSS an opportunity to familiarize itself with the operating environment in which the laminate will be used. Pertinent discussion should elaborate on primary and secondary performance criteria and include a review of conductivity issues, size and geometry, expected design stresses and loading conditions, critical mechanical property requirements and other important design elements, such as appearance, "touch" and "feel", that will directly impact the serviceability of the developed system.

2. Program Objectives: The program objectives should be discussed in detail. This will allow METSS to establish the short and long-term goals of the program sponsor in pursuing the design of the ESD laminate system. Specifically, METSS is interested in prioritizing primary design objectives, properties and features (e.g., charge dissipation characteristics, rigidity, impact resistance, processability, etc.) along with lower priority features that would be considered beneficial if they could be incorporated without compromising system performance (e.g., appearance, repairability, etc.). In this manner, METSS will be able to focus its Phase I program efforts on demonstrating the technical feasibility of incorporating the proposed technologies into the ESD laminate material, while keeping in mind other design requirements that may be further developed under the Phase II program.

3. Review Program Plan: Section E contains a detailed program plan prepared by METSS. The program is designed to address program objectives and demonstrate the technical feasibility of improving the ESD response of laminate worksurfaces, by exploiting recent advances in nanophase materials processing technology, along with copolymer and/or polymer blend technologies to control electrical conductivity and enhance the ESD capabilities of the laminate system. METSS would like to use the meeting to review and discuss critical elements of the program. In particular, METSS is aware that the tiered approach proposed to address the program objectives potentially represents a significant work effort. METSS is eager to fully refine and develop the proposed approach, working closely with the program sponsor to ensure that the program proceeds in the most efficient manner possible. While METSS does not anticipate major changes to the project methodology or objectives, METSS is eager to use the startup meeting to refine and clarify all aspects of the program. Project milestones and timetables will be discussed in order to solidify the practicalities of the proposed testing procedures.

The start of work meeting will set the agenda for the Phase I SBIR program, establishing preferred lines of communication for information exchange and feedback, and providing the foundation for a solid, mutually beneficial working relationship between METSS and the program sponsor. Through the discussions held during the start of work meeting, and continued interactions with the program sponsor throughout the course of the SBIR program, METSS will be able to achieve the program objectives in the most efficient and cost effective manner possible.

Task 2. Technology Review

Melamine resins are often used for coatings or lamination of top surfaces as their properties may be readily tailored to provide transparency, high stiffness, good abrasion resistance and high gloss. Like most polymers however, melamine resins and laminates develop static charges at the surface and throughout a small surface layer when placed in sliding contact with other surfaces, such as equipment and components. Static charge can attract dust and dirt, marring the glossy, clear appearance of the surface, as well as providing the potential for ESD. ESD events can be so small in severity as to be essentially undetectable, but in more severe cases can range from being a minor annoyance to presenting a significant risk to personnel and equipment.

There are two general approaches to generating antistatic behavior; namely, the use of (1) intrinsically conducting polymers and (2) antistatic additives. Efforts to produce intrinsically conducting polymers have focussed principally on improving matrix chemistry, but such polymers are typically costly and difficult to process. Recently, there have been some attempts to polymerize some conducting precursors in a polymeric media, thereby producing conducting composites, which exhibit the intermediate conductivity required in antistatic coatings. Some ionic polymers also act as antistatic materials. The more frequent approach used to generate antistatic behavior involves the use of conducting additives. The conductivity in this case depends on the method of preparation, aspect ratio, hollowness in gross configuration and the critical concentration required to produce a tunneling/percolation effect. In this respect, fibers often perform better than fillers of spherical geometry, due to their higher aspect ratio and ability to overlap conduction bands, leading to higher conductivity at lower loading. Without careful control however, the use of conductive additives in the formulation of a resin layer may result in loss of some desired properties, such as gloss and smoothness, and may interfere in the curing reaction. A substantial amount of information on commercially available antistatics exists in the current literature, particularly due to the high incidence of use of antistatic materials for electronic packaging applications. It should be possible to select appropriate materials and technology required to develop antistatic characteristics on the melamine surface.

Antistatic top coatings and laminates have been formulated with a considerable degree of success using a variety of conductive particle and fiber additives including conductive carbon black, metal salts and oxides, among others.[1,2,3,4,5,6,7,8] This approach has occasionally led to shortcomings in the performance of the coating/laminate in terms of decreased transparency, diminished weathering resistance, inferior mechanical properties (including embrittlement, decreased flexibility and poor processabililty), and sensitivity to temperature and humidity. Additionally, laminates with the degree of particle/fiber loading necessary to provide the requisite degree of conductivity/antistatic performance are often susceptible to

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[1] N. Hardwick, "Controlling ESD via polymer technology" *Advanced Packaging* Sept/Oct 1998, pp 28-32, 1998
[2] V. McGinnis, D. Mangaraj, E. Brooman, R. Schwerzel and K. Hughes, "Electrically conductive polymers: Development and application trends" *Battelle Technical Inputs to Planning, Report No. 77*, Battelle Memorial Institute, Columbus OH, 43201, 1992
[3] G. Grosheim and A. Wiebe, "Electrically conductive laminate", *US Patent No. 4,472,474 1984*
[4] D. Cannady, Jr, "Antistatic laminates containing long carbon fibers" *US Patent No. 4,540,624 1985*
[5] G. Berbeco "Fibrous sheet material for conductive high-pressure laminate" *US Patent No. 4,589,954 1986*
[6] I. Ungar, R. O'Dell, A. Simon and J. Lex, "Static dissipative laminate for work surfaces" *US Patent No. 4,784,908 1988*
[7] C. Wyche, R. O'Dell and I. Ungar, "Static dissipative laminate containing stainless steel fibers" *US Patent No. 5,244,721 1993*
[8] R. O'Dell, C. Wyche and I. Ungar, "Static dissipative laminate containing an intereior special core layer containing carbon fibers" *US Patent No. 5,275,876 1994*

particle removal from dusting of the conductive particles or fluffing of conductive fibers as the laminate wears. Removal of the conductive phase from the surface of the laminate can cause local areas on the surface to become insulative, thereby leading to local static buildup on the surface and the potential for ESD events. Problems have also developed in the past from delamination of the conductive laminates arising from poor adhesion due to the presence of the particles or fibers. Hence, it is desirable either to refine the practice of incorporating conductive additives, or develop intrinsically semiconducting polymers or polymer blends based on melamine oligomers.

Recent advances in materials processing technology, allowing precursor materials to be milled down to nanophase powders, provide the opportunity to make significant progression in the production of high efficiency integrated conductive resin systems. Nanophase materials offer a number of critical advantages over coarser materials in applications such as this where interfacial properties and inter-particle spacing are important, the most obvious of which are the significantly enhanced surface area to volume (SAV) ratio and ability to achieve a high level of powder dispersion at relatively low loading levels. SAV ratios in nano-phase materials are 3 to 4 orders of magnitude greater than those inherent to micro-phase materials, with corresponding improvements in efficiency of interface-critical processes that allow the particle loading to be kept at lower levels for an equivalent level of effect. Maintaining the volume fraction of additives at lower levels has additional benefits in cost minimization and preservation of mechanical properties (there is less perturbance of the matrix microstructure, due to small particle size).

Depending on the molecular level interactions and compatibility between the resin matrix and particle additives, the use of nanophase additives should make it possible to enhance structural properties of the resin through mechanisms of dispersion strengthening, matrix reinforcement and crack blunting. Essentially, a well-compatibilized particle/resin system may itself be expected to behave as a nanocomposite, with the nano-phase additives providing a mechanism of reinforcement and impact resistance to the resin matrix. Nano-phase particle additives also allow for reduced signal attenuation, as the small particle size is less likely to interact with incident wave energy, such as visible light. Specifically, METSS has experience in the use of nanophase additives for other applications in which the matrix polymer material remains completely transparent for high particle loadings, with only a slight haze present.

METSS is working closely with a new source of nanophase materials, which will be used as the main constituent of the conductive additive system in the melamine resin top-layer material. Initially, METSS will concentrate on nanophase carbon[9] additive systems based on particles and fibers, but will also investigate incorporation of metallic nanopowders as dictated by component miscibility and processability. If required, METSS will also investigate the possibility of employing a hybrid additive package, as dictated by program developments.

METSS has examined proprietary coated mica and carbon black fine particulates with a series of resins, including a melamine-formaldeyde polymer on a paper substrate. In preliminary experiments, we have obtained ~20 Ωcm resistivity with a 50% carbon black loading for both a 37 micron thick film of thermosetting acrylic and a 150μm thick melamine-formaldehyde polymer on paper substrate. Thus, the preliminary results indicate that there is more than enough conductivity to dissipate static charge. It should be emphasized that the results obtained relate to micron sized powders, for "proof of concept" purposes only. None of the preliminary work has been optimized and significant improvements in terms of the specific conductivity levels, loading levels necessary, mechanical properties and laminate longevity would be expected from the nanophase equivalents.

[9] Note - The size of the carbon phase additives will ensure transparency.

METSS believes that the use of nanophase materials as conductive additives offers the opportunity to control the static dissipative properties of the laminate to precisely controlled levels, by strategic manipulation of particle loading, particle size, aspect ratio (spherical particle vs. fiber) and particle distribution.

Specifically, the use of nanophase materials would be expected to provide a more uniform, fine dispersion of conductive particles, thereby providing a more homogeneously conductive laminate and bestowing the necessary conductivity to facilitate charge dissipation in local surface features, such as microscopic peaks and ridges on the laminate surface. In traditional particle-containing laminates, such features are typically too small to contain enough conductive phase to dissipate charge efficiently. Therefore, unless one of the contacting surfaces is sufficiently compliant to allow full contact across the static interface, the actual contact area is significantly reduced and the performance of the antistatic surface material suffers accordingly. The use of nanophase conductive materials will alleviate this problem to an appreciable degree, by providing a more uniformly distributed, finely dispersed array of conductive particles that are sufficiently small to uniformly occupy all domains throughout the conductive laminate, including microscopic peaks and ridges on the laminate surface. Additionally, particle removal (dusting and fluffing) due to sliding contact on the worksurface would be significantly reduced with nanophase additives, as the orders of magnitude increase in SAV for nanophase materials would ensure greater contact area between particles and matrix, and therefore greater overall particle adhesion. METSS expects that optimization efforts under the proposed Phase I program would enable the range of successful additives to be extended well beyond carbon into other systems.

The use of nano-phase technology effectively multiplies the efficiency of these materials as conductive additives, thereby decreasing the volume (and weight) of materials that must be added for an equivalent level of performance. This task will focus on identification of materials, in addition to those already identified, which may be used as additives in conductive resin systems, and will target program efforts into a strategically selected group of resin/additive systems to maximize the probability of success.

An alternative approach to providing the requisite ESD protection (and the second tier to METSS' proposed approach) involves the use of polymer blend technology. The easiest route through which to address this is to blend melamine resins with intrinsic semiconducting polymers. Recently, a number of semiconducting polymers, such as polypyrrole (PPy), polythiophene, polyaniline (PA), polyphenylene (PP), etc. have become commercially available. Once doped with a suitable material, such as arsenic pentationide, they provide high conductivity, typically of the order of 10^{-1} S/cm. These materials can be blended with melamine prepolymers, such as trimethyl melamine, to provide a transparent blend with conductivity in the region of 10^{-5} to 10^{-6} S/cm. Acrylic polymers and styrene-acrylate copolymers are quite suitable for the purpose, since in addition to exhibiting antistatic behavior, these polymers can provide resistance to UV degradation.

Intrinsically conducting polymers and polymer blends are inherently fragile and difficult to process. Recent work[10] has attempted to address this problem by formulating core-shell polymers where a core of thermoplastic poly(methyl methacrylate) (PMMA) is coated with a shell of intrinsically conducting PPy. In this manner, the ease of processing of the thermoplastic component of the composite is combined with the conductive properties of the intrinsically conducting component. Using this system, conductivity values in the range of 1×10^{-9} S/cm to 0.1 S/cm were recorded, depending on the concentration of PPy. The processability of these systems can be further enhanced by blending PPy-coated PMMA particles with uncoated PMMA particles.

[10] M. Omastova, J. Pavlinec, J. Pionteck, F. Simon and S. Kosina, "Chemical preparation and characterization of conductive poly(methyl methacrylate)/polypyrrole composites" *Polymer* 39, pp 6559-6566, 1998.

Thirdly, METSS will investigate providing ESD protection through a mechanism of polymerization and copolymerization. It has been demonstrated that pyrrole monomers can be polymerized in vapor phase in a polymeric media of certain viscosity and subsequently the polymer can undergo crosslinking to provide hard laminates. In particular, pyrrole has been vapor phase polymerized in a crosslinkable polystyrene formulation, resulting in a semiconducting polymer. It is proposed that the same can be done using partially polymerized methylated or butylated melamine.

Since antistatic behavior does not require a high level of conductivity, polymers with ionic groups have lower resistivity than nonionic polymers and can potentially be used for antistatic coating or lamination applications. It is proposed that methylol melamines be condensed with acrylic, maleic or fumanic acid, to give the corresponding unsaturated ester, which can then be copolymerized with an acid monomer to provide the ionic resin. The resulting copolymers containing acrylic acid, malecic anhydride or sulfonated styrene can be semiconducting. Their conductivity can be further increased by adding metallic bases such as Ca, Hg, or Zn oxides or hydroxides. The metallic ions will not only provide higher conductivity, but better mechanical properties, due to ionic crosslinking.

Numerous different combinations of additives, polymers and resin systems exist in the literature, which bestow a level of ESD resistance to certain material systems. Since the operating parameters for materials to be developed under this program are well defined however, the list of materials showing genuine potential for use in the proposed system narrows considerably. METSS has identified candidate additives, blends and copolymer systems, which will be included to address program objectives, but will conduct an intensive focused technology review on antistatic additives, bringing together information from the literature and relevant industry sources, and aimed at identifying viable alternatives to those already chosen. This may include looking at commercially available technologies. [1] For Instance, Nevamar has recently reported the development of an ESD laminate structure that reportedly meets the program requirements. METSS will investigate this development and determine its impact on the proposed program. By using the proposed approach, METSS will ensure that potential candidate materials are not overlooked and that the technology pursued under the Phase I program provides the most likely avenue for program success.

The results of the technology review will be used to compile a finalized group of candidate additive materials, blend systems and copolymer systems for inclusion into the testing and development program. METSS will also use the technology review to identify commercial vendors and manufacturers, who in addition to current program support organizations, could have a role in commercialization efforts for the technology in Phase II of the program.

A recent product development by Nevamar may be of particular interest. Nevamar recently reported the development of a new line of Static Dissipative Laminates. Although Nevamar believes that this product addresses the requirements of the MIL-PRF-87893B performance specification, METSS will incorporate the Nevamar product into the SBIR workplan. Testing the Nevamar laminate will generate baseline data to support product formulation efforts and also provide a critical evaluation of product performance. In the event that the Nevamar product does not perform satisfactorily, the data generated from the SBIR program will provide the opportunity for product refinement and further laminate development.

Task 3. Processing and Fabrication Development

This task will establish processing procedures and methodologies necessary to support fabrication of the laminate material. Since fabrication of the laminate system could be accomplished by a variety of processes, depending on which of the technologies investigated proves most promising, it will be necessary to investigate the influence of the various processing conditions on laminate properties, and therefore establish processing conditions to support scale-up fabrication efforts.

Because of the capacity of the additives to wield such considerable influence over system performance, process variables that influence the manner in which these additives interact with the organic resin will be expected to play a correspondingly significant role in material performance. Several important, inter-related factors applicable to particle, fiber or phase interaction with the laminate system will influence, or be influenced by processing and fabrication elements. These include:

- Particle loading (mass fraction): The mass of additives incorporated into the resin matrix will influence the mechanisms of conduction and the effectiveness of the additives in controlling ESD events. Particle loading will have obvious ramifications for overall resin density and transparency, and will also influence processing variables such as rate and temperature of resin cure.
- Particle/phase size and distribution: These elements will have a significant effect on electrical conduction efficiency and homogeneity of the system. Particle size in additive-filled laminates is directly related to how finely dispersed the particles can be throughout the resin matrix, which is a primary determining factor in additive efficacy for a given mass of additive material (level of effect per unit mass of material). A fine, uniform particle distribution is critical in maintaining chemical, structural and mechanical homogeneity throughout the resin system, while creating stable, uniform conductivity over both the entire surface of the laminate and also within local areas of the laminate, such in microscopic peaks and valleys on the surface. Controlled particle distribution can only be achieved by careful control of process variables. Particle distribution studies performed with high resolution Scanning Electron Microscopy (SEM) will provide information necessary to direct process modifications.

  The size, shape and distribution of the conducting phase in a polymer blend would be expected to wield similar influence over the conductive and mechanical properties of the laminate to that of conductive additives. It may be necessary to add a compatibilizer to assist the dispersion process, so that the particle size of the dispersed phase is very, very small and as such, provides a stable, homogeneous conductive media, does not interfere with light transmission in the visible range, or disrupt the homogeneity of the mechanical properties. To this end, a conducting resin, such as a styrene-acrylate copolymer, whose solubility parameter matches that of the melamine resins should be used in the blending process.
- Overall additive composition (relative amounts of each additive): The additive composition controls the mechanisms and effectiveness of the conductive system. Formulation efforts will be targeted towards determination of the optimum concentration of each additive and/or dispersed phase.
- Compatibility: Compatibility between additive materials and the resin matrix will play a determining role in both the ESD performance and mechanical properties of the resin. Poor compatibility or other flaws or imperfections at a matrix/additive interface under stress may lead to crack initiation or delamination. It will be necessary therefore, to be aware of the controlling parameters (surface chemistry, resin wetting of the particle, etc.) in order to optimize compatibility during processing. Compatibility and additive particle wetting will be characterized by SEM and thermal analysis. In the case of the polymer blend preparation, METSS will investigate the use of compatibilizers, if necessary, to ensure that the conductive phase dispersion is sufficiently homogeneous and fine as to provide the requisite conductivity, mechanical and physical properties, (transparency, etc.).

Formulations of intrinsically conducting polymer and copolymer laminates and preparations of polymer blends & composites will be performed by METSS personnel at our facility in Columbus, OH. Polymer blends based on the core-shell composite concept can be readily formulated using well-known, simple

synthesis procedures using a PMMA emulsion in the presence of an aqueous $FeCl_3$/PPy solution. Intrinsically conducting copolymers can be crosslinked by adding formaldehyde or paraformaldehyde in the presence of a suitable catalyst. Hexamine, which provides nascent formaldehyde at high temperatures, can be used to crosslink the melamine resin.

Under this task, METSS will not only develop the resin formulations needed to support the program efforts, but it will also fabricate test samples from the resin samples to support product characterization efforts (Task 4).

Task 4. Testing and Evaluation

The primary goal of this task is to evaluate the conductivity and mechanical properties of the laminate resins. Critical tests, which must be performed to assist with material development and qualification efforts, include:

- Conductivity testing: METSS will build a test cell to support conductivity evaluations and product formulation efforts. The cell will be used to evaluate the conductivity of a series of resin test panels in accordance with MIL-PRF-87893B performance specification. Resin test panels will be fabricated in the first instance from melamine-based resin systems containing various particle and fiber additive packages, and the results evaluated against established pass-fail criteria. In the event that this series of panels does not conform to the specifications set forth in MIL-PRF-87893B, METSS will then begin testing polymer blend formulations commensurate with the second tier of the investigation, followed by testing and characterization of panels processed using materials synthesized with intrinsically conductive polymer/copolymer systems. The data generated from testing each of the systems will be used, along with data from structure & morphology characterization and mechanical testing (detailed below) to refine formulations towards a system that conforms to the performance criteria set forth in the workplan.
- Mechanical testing of resin samples: In order to characterize the mechanical properties of the resin materials and establish the extent of any mechanical property degradation induced by incorporation of the additive package and/or blending, METSS will perform a series of standard mechanical tests on the conductive resin panels. Key tests to be performed will generate information regarding tensile properties (ASTM D638 *Standard Test Method for Tensile Properties of Plastics)*, flexural properties (ASTM D790 *Standard Test methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials)* and impact properties (ASTM D3029 *Standard Test Methods for impact Resistance of Flat, Rigid Plastic Specimens by Means of a Tup (Falling Weight)*) of the resin. METSS will also evaluate adhesion strength between the resin and substrate materials, which relates to delamination failure. Additionally, METSS will test the effect of abrasion on antistatic properties of the laminates to determine the resistance of the laminates to particle dusting and fiber fluffing. Tensile, flexural and impact properties will provide information on particle cohesion and general structural integrity within the resin, and are of direct relevance to the structural properties required in service.

Data generated from testing endeavors will be used where applicable, as feedback information to support improvements in formulation and fabrication methodologies for the laminate prototype. In this manner, the conductive resin system will be continually refined and optimized in order to meet the program goals.

Task 5. Prototype Fabrication and Testing

This task METSS will focus on fabrication of a laminate sheet or panel prototype (or prototypes), following determination of best performing systems, which will provide information on the resin

material's effectiveness as an integral component in a workstation laminate panel system. METSS will fabricate a series of laminate panels, based on melamine resin oligomers hot-pressed into a paper substrate, consulting with the program sponsor, commercial vendors and fabricators to ensure that the samples are fabricated in a manner that best approximates commercial processing practices. Laminate fabrications will be performed over a series of curing temperatures at METSS' facility in Columbus, Ohio using a specially constructed sample mold and a press with heated molding platens. Testing will then be performed to evaluate the effect of process parameters on the product properties (conductivity, ESD characteristics, mechanical properties etc.) as outlined for the resin samples under Task 4. METSS intends to use the conductivity and mechanical property data generated throughout earlier stages of the Phase I program, to select conductive resin system and processing conditions required to fabricate a prototype workstation panel with optimized performance characteristics. Prototype panels could be used to generate extensive background conductivity and mechanical property data to assist in Phase II development efforts.

Laminate formulation efforts will build upon the data generated from the resin formulation and testing tasks detailed under Tasks 3 and 4. Prototype laminate formulation and testing will provide valuable information on the ease of scaling up the formulation efforts developed under Task 3 to full-scale laminate production. When METSS feels that a viable laminate structure that meets the property requirements set forth in the MIL-PRF-87893B has been developed, samples of the laminate will be submitted to the Air Force for intitial qualification.

Task 6. Administrative and Reporting

Progress reports will be written and submitted monthly (or as required by the contract) to update the government on progress and program challenges. A comprehensive final report will be submitted at the end of the Phase I program, completely detailing the efforts performed under the Phase I program and the results of these efforts, and demonstrating the technical feasibility of program efforts. The decisions that were made to drive the direction of the program will be reviewed and justified based on available data. Based on the results of the Phase I work, METSS will be able to provide a detailed description of a prototype laminate system for workstation applications that will be further developed and optimized under the Phase II program.

F. RELATED WORK

F.1 Technical Expertise and Experience

The staff of METSS has a great deal of practical and theoretical experience that will be used to support the proposed program, specifically with regard to the following:

- product formulation and development – including polymers, resins, coatings, rubbers and fine-dispersion metallic alloys
- development of specialty polymer blends and composites
- materials/chemical processing, testing, analysis and characterization
- conduction mechanisms in polymer gel electrolytes
- characterization of solid polymer electrolytes
- blending, alloying and compatibilization with specialty additives in polymeric matrices
- polymer, rubber and resin synthesis.

METSS staff posses specific expertise in all areas of the proposed development program. Dr. Kenneth Heater (the proposed PI) has conducted a number of programs of the proposed nature, directing multi-disciplinary teams and lending his expertise in materials science and engineering to direct product development, testing, evaluation and qualification efforts. Other METSS staff members have also worked on numerous projects of this nature, developing and assessing alternative technologies for applications including coatings, fire-retardants, specialty resin additives, particle compatibilization, resin formulation, polymer blend compatibilization and an extensive array of highly specialized polymer testing. Dr. Heater and Dr. D. Mangaraj led or contributed technical expertise to many of these and related efforts, particularly with regard to formulation of specialty coatings, the evaluation of reactive processing of flame retardants, low-profile composite additives, and the design and implementation of testing & evaluation programs to assess product or process performance. Dr. Mark Hodge has worked extensively in a broad range of polymers and composites, including studies on conductive polymer gel electrolytes, specialty additives in resin systems, mechanical property optimization and degradation mechanisms in polymers and light metal alloys as well as broad experience in materials testing and characterization. Dr. Hodge has just started a Phase I program in which the nano-phase additives will be used as fire retardants in advanced resins systems. Dr. Mangaraj has extensive experience in the field of conductive polymers and polymer synthesis and has co-authored several research papers and reports in both fields. Dr. Mangaraj has also worked in the area of polymer synthesis and formulation, with specific expertise in the field of rubber and resin synthesis and formulation.

METSS has proven expertise in conducting strategic, applied product development and product formulation programs. Our experience base includes the development of paints, adhesives, sealants, functional coatings, composite resins, foams, flame retardants, and cosmetic products. Specific efforts include the development of a "child-safe" glue stick product for a major commercial supplier (confidential), room temperature cure structural adhesives for steel, aluminum, and glass fiber reinforced polymer composites, high temperature composite components and the development of conductive sealants for aircraft. Currently, METSS is developing environmentally friendly replacement products for aircraft hydraulic systems, deicing applications, and dielectric fluid applications. METSS is also developing a high temperature/chemical resistant rubber inner-liner (solvent dispersed) for aircraft fuel bladders and advanced packaging materials that incorporate ESD and EMI property characteristics. Our success on these programs supports our ability to conduct programs of the proposed nature.

METSS has an established business relationship with Micronisers, a specialty nanophase materials producer in Australia. Micronisers is the owner of a proprietary process that produces nanophase materials by high intensity milling in various media. The process allows the particle size to be controlled to within accurate limits and is not as energy intensive as other methods of nanophase powder production (e.g. vapor deposition, etc.) and as such, can produce high purity specialty additives in an extremely cost effective manner (typically for less than 1/3 of the cost of equivalent powders produced by traditional methods). Commensurate with the established relationship between METSS and Micronisers, Micronisers has agreed to supply the nanophase additives at no cost to the program, in order to support Phase I development efforts. This supply agreement will enable METSS to concentrate program resources into formulation development and property qualification efforts, thereby accelerating the transition from Phase I proof of concept studies to more stringent development and qualification efforts and commercialization under Phase II.

F.2 Business Interest and Commercialization

Since . METSS has been awarded 11 Phase I SBIR programs. Our success in conducting these programs has been rewarded with 6 Phase II programs, including five ongoing programs and one recent award for aircraft fuel bladder materials (Table 2). Two of the Phase I programs are recent awards. Two of the Phase I projects which were not moved toward Phase II funding were determined not to be

commercially viable by METSS. The remaining program was not pursued after an internal reorganization of the sponsoring agency. In addition to its SBIR program efforts, METSS is currently conducting two similarly structured programs for a private utility company for the development of *Biodegradable, Direct Replacement Dielectric Fluids for (1) Underground HV Cables and (2) Transformers.* METSS is also conducting a product development effort for the United Soybean Council to develop commercial dry film lubricants based on soybean oils and derivatives. METSS is actively supporting the commercialization of all of the SBIR programs (see Section H). Our success in these programs has been supported by our expertise in conducting materials development programs and are focus of using applied technologies to support project development efforts.

Table 2. Ongoing Phase II SBIR Programs

Title	Contract No.	POC
Barium Free Environmentally Friendly Corrosion Inhibitors	F33615-96-C-5074	Ed Snyder, AFRL/MLBT (937) 255-9036
Biodegradable, Direct Replacement Hydraulic Fluids	F33615-97-C-5008	Ed Snyder, AFRL /MLBT (937) 255-9036
Recycling of Polymeric Aircraft Transparencies	F33615-96-C-3405	Richard Smith, AFRL /FIVE (937) 255-6078
Environmentally Benign Aircraft Deicing/Anti-Icing Fluids	F33615-98-C-5650	Steven Anderson, Capt., AFRL/MLQ (850) 283-6039
Adhesive Sealable Barrier Material	F30600-98-D-0052	Susan Evans, AFMC LSO/LOPM (937) 257-7445
High Temperature Resistant Aircraft Fuel Bladder Materials	Under Negotiation	Allan Fletcher, AFRL/MLSE (937) 255-7481

A brief description of the technology commercialization efforts associated with some of these efforts is presented in Table 3. The commercialization strategy for the current program is likely to follow similar lines as the efforts described. However, the actual commercialization strategy that will be employed to ensure transfer of the technology developed under the SBIR program is strongly dependent on a number of factors and could take one of several forms. The drivers behind which commercialization path is taken will be defined largely by the candidate materials meeting the program requirements and the commercial availability of these materials. In any event, METSS will perform whatever efforts are necessary to ensure the technology developed under the SBIR program is available to support subsequent product commercialization efforts.

G. RELATIONSHIP WITH FUTURE R&D

The Phase I program efforts will set a very solid foundation for Phase II development efforts, related product development efforts, and product commercialization. Under the Phase I program, METSS will strive to develop a series of conductive laminate systems representative of the various additive, blend and/or copolymer systems identified under Task 2 within the parameters of the Phase I time and budget constraints. Due to the required processing and testing efforts necessary to support qualification of the different resin formulations however, it is anticipated that development efforts will most likely produce no more than two or three best-performing products. METSS has intentionally designed a series of Phase I tasks aimed at establishing the technical feasibility of blending nanophase additives into a resin system along with other additives to produce an integrated ESD compliant system. These efforts will allow METSS to broaden the scope of the proposed approach in subsequent efforts to further develop this technology and address other issues, such as those relating to manufacturing scale-up, appearance, etc. in

greater detail under the Phase II program. Emphasis on these efforts and defined tasks will allow METSS to gain a better understanding of the potential products that may be developed using the proposed approach and the effort required to fully develop these products (information that will be needed to support the Phase II proposal efforts). In addition, this information will support market development activities and may impact product commercialization plans.

Table 3. Current Commercialization Efforts

Lubricants - METSS has established a joint venture with an existing lubricant manufacturing company to pursue commercialization of dielectric fluids and is currently negotiating an exclusive license from an industrial chemical supplier to use certain chemistries for this application. A marketing group established by the sponsoring agency will assist commercialization of this technology. METSS' partner in this joint venture is also interested in assisting METSS in the commercialization of biodegradable hydraulic fluids and environmentally friendly corrosion inhibitors. Similar interest has been expressed by an Oklahoma based raw materials producer, who has made an offer to purchase part of METSS to obtain access to these technologies.
Recycling - METSS is pursuing the commercialization of three processes related to the polycarbonate (PC) recycling program: (1) an environmentally friendly process for recycling high valued PC resin; (2) an environmentally friendly process for removing protective coatings from PC scrap; and (3) the production of a specialty chemical as a by-product from the coatings removal process. METSS is currently discussing items 1&2 with GE and is seeking support in terms of a licensing agreement or a purchase contract for recycled product. Item 3, the specialty chemical production, has just been initiated but has already found support from industry and investors who are willing to support this process if it is determined to be commercially viable.
Deicing - The Phase II deicing program is moving rapidly toward product commercialization. METSS is currently in negotiation with the only US supplier of one of the key components in the deicing formulations under development by METSS to pursue commercialization of METSS' deicing technologies. METSS is working with this company to initiate product testing of roadway and landscape deicing products in the winter of 1998. In addition, METSS has initiated discussions with an existing deicer supplier to use METSS technology to address conformance testing problems they are experiencing with their deicing materials. The remaining program is too early in its development to pursue commercialization. However, METSS expects to use strategic teaming methods to facilitate commercialization of technologies developed as a result of these program efforts.

H. COMMERCILIZATION STRATEGY

Under Section F.2, a description was provided on current product development activities and the manner in which METSS is seeking to commercialize these technologies. As noted, METSS is making a conscious effort to move commercialization efforts outside of our R&D business. The management of METSS made this decision for the simple reason that METSS was formed, structured and staffed to provide the best mix of technical talents to conduct successful R&D programs and rapidly develop new products and processes for commercial markets, but not to manufacture and distribute products. We plan to keep our focus on our strengths by transitioning technologies out of METSS once they have matured to the point where they are ready to be integrated into a business focused on commercialization. This strategy will allow METSS to continue to focus on our strength of developing new products and technologies, and eventually grow to the point where returns from outside commercialization ventures will allow us to proactively develop products for new markets, without relying on contract support dollars to fuel our growth in these new areas.

The current commercialization strategy for the development of conductive laminate systems for workspace applications developed under the proposed program will involve the formation of a business relationship between METSS Corporation and a manufacturer/supplier/fabricator to be determined during the Phase I program. The exact nature of this relationship (e.g., joint venture, licensing agreement, OEM operation, etc.) will to a large extent be determined by factors such as:

- The significance of technical achievements accomplished under the proposed program.
- The relative contributions made by each party.
- Market potential for the products developed.
- The relative role of each party in marketing and developing sales streams for the products.

In the simplest of terms, METSS will provide the technology around which this business relationship will be built and throughout the Phase I program effort, will strive to identify avenues through which the resources needed to facilitate product commercialization may be provided. METSS wants to maintain its core function as an R&D provider, an area in which we have been extremely successful to date. We anticipate that the technology developed throughout this program will place us in a position to forge a number of business relationships with fabricators and manufacturers of laminate structures. The technology developed under the Phase I program does not however, restrict METSS to forming relationships with laminate fabricators. The technology should be readily applicable to other commercial areas, such as packaging, flexible ESD mats and sheets, and a variety of other applications.

The relationship with Micronisers has positioned METSS to be a supplier of both raw materials (through a prior royalty-based agreement with Micronisers) and technology. This provides METSS with a great deal of flexibility from which to pursue commercialization alliances with a variety of companies in different industries, where the relationship in each case can be driven by the technology and the circumstances of each company. For example, METSS could pursue a relationship on the basis of licensing additive package technology to established manufacturers, to allow them to achieve greater market penetration by developing a superior product (such as that envisaged as possible with Nevamar). In addition to this, METSS could act as a supplier of raw material components for the additive package to enhance an existing product or process. This scenario is seen as the most likely occurrence, as it offers the most potential for ready integration of the technology into established processes and markets, although METSS is extremely flexible in the manner in which we define our commercialization partnerships.

The majority of the SBIR program dollars will be used to support product development efforts, address processing and fabrication issues that may affect product performance, and qualify the products developed against the performance specifications. METSS will perform the product development efforts and work interactively with commercial suppliers and fabricators identified during the Phase I program effort in order to address production and fabrication issues. Market potential of the products and processes developed in association with the SBIR program will be assessed in conjunction with such suppliers and fabricators and a strategy for commercial implementation of the products and processes developed around respective experience and past successful business strategies.

I. KEY PERSONNEL

METSS is fortunate to be staffed with a number of scientists and engineers with extensive backgrounds in engineering, materials science, and chemistry. METSS is currently staffed by 15 employees, of which have 5 Ph.D.'s in chemistry, materials science, or engineering. As a group, the senior staff of METSS has a significant amount of practical experience in developing and testing product formulations for a number of

different industries, including composites, coatings, adhesives and fluids. The laboratory staff of METSS consists of qualified and trained technicians, each with BS degrees in chemistry or chemical engineering and each with experience in formulation, testing and characterization. Each member of METSS staff will contribute to the program in some manner over the course of the program. However, the following staff members will be primarily responsible for the day to day operations of the program, to ensure that each task is carried out as planned and the objectives of the program are met.

Dr. Kenneth J. Heater will serve as the principle investigator on the proposed program, leading and coordinating the program efforts. Dr. Heater holds a B.S.E. in Mechanical Engineering, a M.S. in Materials Science, and a Ph.D. in Materials Science from Duke University. Dr. Heater's engineering background brings significant practical experience with regard to testing and characterization of materials, with specific experience designing test protocols to evaluate new materials and direct materials development efforts. Dr. Heater's primary expertise lies in the area of polymeric material systems – including polymer matrix composite materials, specifically structure/property relationships, fracture, failure and degradation, processing and specialized analysis and spectroscopy techniques. Dr. Heater has over 10 years of experience in polymeric and composite materials. His dissertation work, *A Positron Annihilation Study of Free Volume and Structural Relaxation in Physically Aged Polycarbonate*, was funded by the National Science Foundation to further the advancement of PAS techniques. After completing his graduate studies, Dr. Heater served as a Principal Research Scientist in the Polymer Science and Technology Group at Battelle Memorial Institute (Columbus, Ohio), where he led all positron spectroscopy related research and development efforts and provided major contributions to polymer R&D efforts. Dr. Heater is currently Vice President and a Senior Engineer of METSS Corporation. As an owner of METSS, Dr. Heater is personally involved in all of METSS R&D programs and works interactively with the staff of METSS to make sure project efforts are moved toward commercialization in a timely manner and that the work performed in support of these efforts is of the utmost quality. He is currently directing R&D efforts for the development of rust inhibited aircraft hydraulic fluids using non-heavy metal free corrosion inhibiting technologies, the development of high temperature/chemical resistance rubber inner liner materials for aircraft fuel bladders, and the development of high performance packaging materials for DoD and commercial applications. Dr. Heater's resume is included at the end of this proposal.

Dr. R. Mark Hodge will work closely with Dr. Heater to support the program technical objectives and commercialization efforts. Dr. Hodge holds an Honors degree in Materials Engineering and a Ph.D. in Materials Engineering from Monash University (Australia). Dr. Hodge has extensive materials fabrication, processing, testing and characterization experience, and has worked on a number of materials development, processing and characterization projects, especially in the area of polymeric materials. His main areas of activity are in polymer, composite and multi-component system processing, testing and characterization, and materials failure and degradation. Dr. Hodge's Ph.D. thesis, entitled *The Influence of Water on Structure-Property Relationships and Morphology in Semicrystalline Poly(vinyl alcohol) Fibers* entailed the study of novel processing techniques, molecular level phase interactions and strain-induced microstructural changes during the production of high strength and stiffness films and fibers. Dr. Hodge held a faculty position at Monash University during the final year of his graduate studies – the first and only person ever to do so in the Department of Materials Engineering. He has also participated in research projects in the area of high-temperature, abrasion resistant isotropic carbon coatings, and fine-grained, dispersion strengthened alloys and published research in the area of ionic conductivity in polymer electrolytes. Dr. Hodge was an active member of the Australian Cooperative Research Center for Polymer Blends and has managed a number of research programs for commercial and government organizations and has a number of published research works in international journals and conference proceedings.

Dr. D. Mangaraj will bring extensive resin and polymer formulations experience and additive compatibilizations expertise in the field of conductive polymers to support program efforts. He has a Ph.D. in Polymer Science from the Manchester University (UK). Since graduation, he has worked on polymer product and process development in many areas, including composites, resins and rubbers. His main involvement in resins is in the area of system compatibility, conductive additives, low profile additives, reactive processing and flame-retardants. He has co-authored a major technology review, entitled "Electrically Conductive Polymers; Development and Application Trends", along with multi-client reports on compatibilization and low profile additives. Additionally, he has published articles on the use of chemical compatibilization in recycling of plastics waste. He has also written a report on thermodynamic models of polymer-polymer miscibility and has broad experience in selecting and applying materials in multicomponent systems to assure compatibility and enhance material performance.

J. FACILITIES AND EQUIPMENT

METSS will provide all of the equipment, materials, and facilities needed to accomplish the goals of the Phase II SBIR program. The facilities of METSS consist of over 6500 ft^2 of office and laboratory space located in Columbus, Ohio, specifically designed to support METSS' R&D efforts. METSS' facility is compliant with all applicable local, state, and federal regulations (environmental, health, and safety). METSS is also a member of the *Center for Advanced Polymer and Composites Engineering* (CAPCE) at Ohio State University. Membership provides METSS access to all of the analytical, instrumentation, and support facilities of The Ohio State University on an "at-cost" basis. This includes access to advanced analytical equipment such as NMR and GS-MS, electron microscopy, physical property test equipment, and composite processing equipment.

Specific capabilities available to support the proposed program include:

- *Thermal analysis instrumentation, including TGA, DSC, DTA and Dielectric Spectroscopy.*
- *Instron testing machines and Flexometers*
- *FTIR (with near, mid, and far infrared capabilities)*
- *Extrusion, spraying, blowing and pouring equipment*
- *Mixers, compounding equipment, and application equipment*
- *Ovens, furnaces and capabilities to support flammability testing*
- *Optical and electron microscopes, and metallography equipment*
- *Vacuum bags, pumps and fittings to support VARTM processing.*

K. CONSULTANTS

No consultants will be required to support the objectives of the Phase II program efforts.

L. PRIOR, CURRENT, OR PENDING SUPPORT

There is nor prior, current, or pending support for the proposed work.

Vice President / Senior Engineer

Education

B.S.E., Mechanical Engineering, Duke University
M.S., Materials Science, Duke University
Ph.D., Materials Science, Duke University

Qualifications

Dr. Heater's primary expertise lies in the area of Positron Annihilation Spectroscopy (PAS), a nondestructive technique for materials evaluation, especially suited for monitoring physical changes and degradation processes in polymer-based systems. His dissertation work, "A Positron Annihilation Study of Free Volume and Structural Relaxation in Physically Aged Polycarbonate", was funded by the National Science Foundation to further the advancement of PAS technology. His experience with PAS includes significant knowledge of the system electronics and software development, the application of PAS techniques to materials characterization, the interactions of positrons in materials, and free volume theory in polymers.

Dr. Heater utilizes his background in engineering and materials science to provide technical expertise and support in a number of different capacities. Dr. Heater has served as a program manager for a number of government and industrial programs, providing technical support and direction for materials research and development programs, and programs requiring materials selection, testing, and evaluation. Dr. Heater has significant experience in materials testing and characterization, and liquid-polymer interactions. He has been active in providing support to the U.S. Army and the U.S. Air Force for materials applications in hazardous environments, and has served as an active member of the Test Methodology Subgroup of the Nuclear, Biological, and Chemical Contamination Survivability (NBCCS) Task Working Group (TWG). Dr. Heater's background in engineering and materials characterization complements his interest and knowledge of degradation processes in polymers, and failure analysis of polymer systems.

Relevant Experience

Dr. Heater's experience includes:

- characterization and modeling UV and gamma irradiation damage in polymers
- characterization of physical aging and structural relaxation in polymers
- the application of thermal and mechanical methods of polymer characterization including Instron testing, DMTA, DSC, DTA, TMA, and TGA
- material applications in hazardous chemical environments
- mechanical degradation of materials in hazardous environments
- liquid-polymer interactions: chemical and physical effects, diffusion mechanisms, polymer solubility, predictive modeling, supercritical fluids processing
- test methodology development for assessing the effects of hazardous chemicals on the physical and mechanical properties of materials
- selection and evaluation of materials for various applications, products, or industrial processes

- the application of structural and insulating polymer materials in cryogenic applications
- technical consultation and research, failure analysis, and mechanical testing
- the manufacture, application, and cure of powder coatings
- the application of infrared radiation ovens in the coatings industry
- the development and fabrication of fiber optic sensors for chemical and vibration analysis

Prior Professional Experience

Principle Research Scientist (Battelle Polymer Center), Battelle Memorial Institute, Columbus, Ohio; Sept. 1991 Dec. 1993; Lead Positron Annihilation Spectroscopy Program R&D efforts; served as program manager for government and industrial programs, providing technical support and direction for materials research and development programs, as well as programs requiring materials selection, testing, and evaluation; CPRP certification for U.S. Army chemical defense work; Polymer Center liaison to Battelle's Hazardous Materials Research Facility; provided to materials support to other groups in Battelle doing chemical defense work.

Consulting Partner, Technical Consultation Resources, Durham, N.C.; June 1989 - Sept. 1991; Technical consultation and research, failure analysis, finite element analysis and modeling for accident reconstruction, and materials characterization and testing.

Graduate Research Assistant, Duke University, Durham, N.C.; Sept. 1987 - Sept. 1991; Vax systems manager, maintenance and operation of positron annihilation lifetime spectroscopy systems, thermal analysis equipment, and Instron testing equipment.

Graduate Research Chemist, Becton Dickinson & Company Research Center, Research Triangle Park, N.C.; Sept. - Dec. 1988; Physical and mechanical characterization of polymer materials for use in biological applications.

Product Engineer, Westinghouse Electric Corporation, Sumter S.C.; June - Sept. 1987; Inside sales contact for panelboards and switchboards: price quotations, technical assistance and support, product design for fabrication.

Student Engineer, Santee Cooper Electric Cooperative, Moncks Corner, S.C.; June - August 1986; Evaluation of infrared scanning thermography equipment as a means of determining heat loss in power generation stations; software development for heat loss analysis.

Affiliations and Honors

Dr. Heater is a member of the American Society of Mechanical Engineers, American Society for Testing and Materials, The New York Academy of Sciences, Materials Research Society, Federated Society of Coatings Technology, and Pi Tau Sigma Honorary Engineering Society.

Dr. Heater has received the following honors:

- Battelle Key Contributor Award for Leadership and Key Client Development, 1992
- National Science Foundation Fellowship, August, 1987-January, 1991
- Student Presentation Competition Winner, Materials Research Society, Nov., 1989
- Engineering in Training, North Carolina Registration #A11406
- Raymond C. Gaugler Award in Materials Science and Engineering.

Patents

United States Patent - Patent Number 5,451,457; September 19, 1995; *Method and Materials for Protecting Glass* - Patent encompasses the use of polyethylenes, having a molecular weight of at

least 100,000, as a material to separate and protect glass sheets from damage and marring during shipping, and facilitate easy separation of stacked glass sheets by automated methods.

United States Patent - Patent Number 5,609,924; Mar. 11, 1997; *Method for Protecting Glass* - Patent encompasses the use of polyethylenes, having a molecular weight of at least 150,000, as a material to separate and protect glass sheets from damage and marring during shipping, and facilitate easy separation of stacked glass sheets by automated methods.

Selected Publications

A. J. Hill, K. J. Heater, and C. M. Agrawal, "The Effects of Physical Aging on Polycarbonate", *J. Polym. Sci.:Polym. Phys. Ed.*, 28, (1990) 387.

C. M. Agrawal, K. J. Heater, and A. J. Hill, "Physical Properties of Aged Polycarbonate as a Function of Cooling Rate", *J. Matter. Sci. Lett.*, 8, (1989) 1414.

K. J. Heater and P. L. Jones, "On the Characterization of Glassy Polymers by Positron Annihilation Lifetime Spectroscopy", *Nuclear Instruments and Methods in Physics Research,* 1356/57 (1991).

K. J. Heater and P. L. Jones, "Free Volume Relaxation in Polycarbonate as a Function of Physical Aging", *Proceedings, Materials Research Society,* Vol. 215 (1990) p 207.

Kenneth J. Heater and P. L. Jones, "The Utility of Positron Annihilation Lifetime Spectroscopy in Characterizing the Molecular Response of Polymers," *Proceedings, ASTM F-7 Meeting on Aircraft Transparency Materials,* Arlington, Texas, October 1991.

Kenneth J. Heater and William F. McDonald, "Positron Annihilation Lifetime Methods for Evaluating Moisture Absorption in Polymers", *Proceedings, 1992 ASNT Spring Conference* Orlando, Florida, March 1992.

R.J. Dick, K.J. Heater, V.D. McGinnis, W.F. McDonald, and R.E. Russell, "Comparison of the Effectiveness of Electric IR and Other Energy Sources to Cure Powder Coatings", *J. Coatings Technology*, April, 1994.

Kenneth J. Heater and William F. McDonald, "A Review of Positron Annihilation Lifetime Spectroscopy and Its Application in Coatings", *Proceedings of the 36th Annual Technical Symposium, Cleveland Society of Coatings Technology, Cleveland, OH, May 1993.*

Donald M. Bigg and Kenneth J. Heater, "Aging of Polycarbonate Transparencies", *Proceedings, Conference on Aerospace Transparent Materials and Enclosures*, San Diego, CA., August 1993.

K. J. Heater and G. Salee, "A Positron Annihilation Lifetime Spectroscopy Study of Gamma Radiation Induced Degradation in Polymer Materials", *39th International SAMPE Symposium and Exhibition*, Anaheim, CA, April 1994, Invited Paper.

P.M. Randall, A.B. Parsons, and K.J. Heater, "Evaluation of Supercritical CO2 Spray Technology as a Cost Effective Approach to Reduction of Solvents in Wood Finishing", *AICHE 1994 Summer National Meeting*, Denver, Colorado, August 1994.

K. Heater and D. Tomasko, "Processing of Epoxy Resins Using Carbon Dioxide as an Antisolvent", *J. Supercritical Fluids*, in review.

CSIRO
AUSTRALIA

Manufacturing Science and Technology
Normanby Road, Clayton, Vic. Postal Address: Private Bag 33, Clayton South MDC, Vic. 3169
Telephone (03) 9545 2777, Fax: (03) 9544 1128

Dr. R. Mark Hodge
Principal Research Engineer
METSS Corporation
720G Lakeview Plaza Blvd
Columbus OH 43085
USA

Friday,

Ref: ***SBIR AF99-146: Development of Static Dissipative Hard Laminate Surfaces***

Dear Mark,

Further to our discussions and your visits to our facility, we would like to confirm both the CSIRO's and Micronisers' interest in your SBIR program and reiterate our desire to support your Phase I program efforts.

Micronisers produces transparent metal oxides, coated particles, metal particles, nucleating and clarifying agents for a variety of pharmaceutical, cosmetic, veterinary, medical and agricultural products, as well as specialized pigments and ink jet inks. The CSIRO provides the R&D infrastructure to Micronisers for new product and process development on a wide front. We believe our current technology gives Micronisers a substantial advantage in nanophase particulates.

Your proposed use of nanophase additives for conductive applications is of great interest to Micronisers and CSIRO. We will be delighted to assist you in achieving your Phase I program goals by providing a reasonable number of nanophase additives to you at no cost to the proposed program, to support your preliminary formulations efforts. This should allow you to focus your efforts and program dollars into methodology development and should mean that your program data will translate more readily to complement ongoing efforts at American Foam Technologies.

We see this as a program of mutual benefit, with the potential to develop new markets for these materials in a number of industries, and we are confident that the potential to transition the technology developed under the proposed program into viable commodity products is high. We will be most interested in taking a larger role in your program as it progresses beyond the proof of concept stages.

Mr Michael Bos, the owner and General Manager of Micronisers, is currently on vacation. However, I have discussed the contents of this letter with him by phone and have his full concurrence. Please let me know if we can be of any further assistance with developing the formulation and testing methodologies and keep us appraised of your progress. We look forward to the potential of interacting with you under the SBIR program.

Kind regards

Dr TW Turney
Leader, Particulates Processing
CSIRO

And on behalf of Michael Bos,
Managing Director,
Micronisers Pty Ltd.

SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM
COMPANY COMMERCIALIZATION REPORT
Failure to fill in all appropriate spaces may cause your proposal to be disqualified

FIRM NAME: METSS Corporation

MAIL ADDRESS: 720 G Lakeview Plaza Blvd.

CITY: Columbus STATE: OH ZIP: 43085

- How many Phase II SBIR or STTR awards has your firm received from the Federal Government (including DoD)? (The answer "none" will not affect your ability to obtain an SBIR award.) 5
- If your firm has received 5 or more Phase II SBIR and/or STTR awards from the Federal Government and the first award was received prior to Jan. 1, 1991, what percentage of your firm's revenues during your last fiscal year is Federal SBIR and/or STTR funding? N/A
- Identify each Phase II SBIR and/or STTR project your firm has received and, for each project, provide the total revenue to date from resulting sales of new products to DoD or its prime contractors, other government agencies, and private sector customers. Also provide total non-SBIR, non-STTR funding received from government and private sector sources to further develop the SBIR technology (including R&D, manufacturing, marketing, etc.). Apportion sales revenue and non-SBIR, non-STTR funding among the various Phase II projects without double-counting. (See back for further instruction.)

Agency: USAF Topic Number: AF95-173 Contract Number: F33615-96-C-5074
Project Title: Environmentally Compliant Corrosion Inhibitors for Aerospace Materials
DoD/Primes Sales: * Other Gov't Sales: * Private Sector Sales: *
non-SBIR/STTR Gov't Funds: * non-SBIR/STTR Private Sector Funds: *
* Program in Progress - No sales at this time

Agency: USAF Topic Number: AF95-149 Contract Number: F33615-95-C-3403
Project Title: Products to Reduce Aircraft Transparency System Cost of Ownership Recycling of Polymeric Aircraft Transparencies
DoD/Primes Sales: * Other Gov't Sales: * Private Sector Sales: *
non-SBIR/STTR Gov't Funds: * non-SBIR/STTR Private Sector Funds: *
* Program in Progress - No sales at this time

Agency: USAF Topic Number: AF96-161 Contract Number: F33615-97-C-5008
Project Title: Biodegradable, Direct Replacement Hydraulic Fluids for Mil-H-5606 and Mil-H-83282
DoD/Primes Sales: * Other Gov't Sales: * Private Sector Sales: *
non-SBIR/STTR Gov't Funds: * non-SBIR/STTR Private Sector Funds: *
* Program in Progress - No sales at this time

Agency: USAF Topic Number: AF97-173 Contract Number: F33615-98-C-5650
Project Title: Environmentally Benign Deicing/Anti-Icing Technology
DoD/Primes Sales: * Other Gov't Sales: * Private Sector Sales: *
non-SBIR/STTR Gov't Funds: * non-SBIR/STTR Private Sector Funds: *
* Program in Progress - No sales at this time

Agency: USAF Topic Number: AF97-245 Contract Number: F33600-98-C-0052
Project Title: Adhesive Barrier Material
DoD/Primes Sales: * Other Gov't Sales: * Private Sector Sales: *
non-SBIR/STTR Gov't Funds: * non-SBIR/STTR Private Sector Funds: *
* Program in Progress - No sales at this time

FIRM CORPORATE OFFICIAL

NAME: Kenneth J. Heater, PhD TELEPHONE: (614)842-6600

TITLE: Vice-President FAX: (614)842-6601

K. J. Heater
SIGNATURE OF FIRM CORPORATE OFFICIAL DATE (Page ___ of ___)

DEPARTMENT OF THE AIR FORCE
AIR FORCE RESEARCH LABORATORY
WRIGHT-PATTERSON AIR FORCE BASE OHIO 45433

MEMORANDUM FOR METSS Corporation
720 G Lakeview Plaza Blvd.
Columbus OH 43085

FROM: AFRL/MLOP BLDG 653
2977 P STREET STE 13
WRIGHT-PATTERSON AFB OH 45433-7318

SUBJECT: Air Force Small Business Innovation Research (SBIR) Program, DOD Solicitation 99.1

CONTROL NO.: 99ML-015

1. Your proposal, entitled **Development of Static Dissipative Hard Laminate Surfaces** submitted to the Materials and Manufacturing Directorate in response to Topic No. AF99-146 of the subject solicitation has been selected as one the Materials and Manufacturing Directorate plans to award subject to availability of funds and successful negotiations between the AFRL/ML Contracting Division and your company.

2. You may expect to be contacted by the assigned buyer from the AFRL/ML Contracting Division in the future, currently estimated at 30 to 60 days from the date of this letter. Due to the volume of work associated with the SBIR awards, please refrain from contacting the contracting personnel during this timeframe.

3. In accordance with paragraph 3.6 (page 6) of the subject document, Phase II proposals are to be submitted only at the request of the agency. If, at the conclusion of the fifth month of your contract, you are requested to submit a proposal it should be submitted within 30 days after the request is made. This schedule allows sufficient time to evaluate the Phase II proposals, complete the selection process and initiate internal contract procedures. Our goal is to minimize or eliminate any gaps in contractual coverage for Phase II selectees.

Sharon E. Starr
SHARON E. STARR
SBIR Program Manager
Materials & Manufacturing Directorate

DEPARTMENT OF THE AIR FORCE
AIR FORCE RESEARCH LABORATORY
WRIGHT-PATTERSON AIR FORCE BASE OHIO 45433

MEMORANDUM FOR METSS Corporation
720 G Lakeview Plaza Blvd.
Columbus OH 43085

FROM: AFRL/MLOP
2977 "P" Street, Ste 13
Wright-Patterson AFB OH 45433-7746

SUBJECT: Air Force Small Business Innovation Research (SBIR) Program, DoD Solicitation 99.1- Control Number 99ML-015

1. Please be advised that your proposal, entitled "Development of Static Dissipative Hard Laminate Surfaces" submitted in response to the Air Force Small Business Innovation Research (SBIR) Topic AF99-146 for DoD Solicitation 99.1, is under evaluation.

2. We anticipate proposal evaluations to be completed bv the end of February. Your company will be notified in writing by regarding the status of your proposal. Due to the volume of work associated with the SBIR process, please refrain from contacting this office prior to this date in order to determine the status of your proposal. Those proposals selected for funding will be award by

3. Thank you for your participation in the Air Force SBIR Program. If you have any questions, please contact me at (937) 656-9221. Be sure to reference the Control Number assigned to your proposal in the above subject when referring to your proposal.

Sharon E. Starr
SHARON E. STARR
SBIR Program Manager
Materials & Manufacturing Directorate

Memo

To: Dr. Kenneth Heater
cc: Mike Manders, Steve Gerken, Mick Hitchcock
From: Julius Brodbeck
Date:
Re: Analysis of METSS samples 1, 2, & 14 for Top to Top Resistance and Static Dissipation at both 10% and 50% Relative Humidity (RH) and 72 °F.

The following discussion refers to the 5 page attached Excel spread sheet (Sam-1214.xls).

Attention focused primarily on sample 14 since it was large enough (16 x 18") to get sufficient data. This sample did not have a scrim layer and reportedly only the top layer was treated with conductive polymer. The sample had a textured, navy blue, semi-gloss surface. Further description of the laminates and test equipment is on page 1 of the spreadsheet.

Laminate 14 was mounted on ¾" furniture grade plywood using 8 bolts around the edges to hold it flat. Laminates 1 and 2 were glued to ¾ furniture grade plywood. The laminates were isolated from ground during the top to top resistance test (Rtt) and the Resistivity test. The samples were grounded for the static dissipation test.

All three laminates passed the top to top (Rtt) resistance tests at 50% relative humidity (RH). Refer to page 4. Only the sample 14 was tested for Rtt at 10% RH see page 2. In both Rtt tests (10 & 50% RH) on sample 14, the variation of resistance with distance between the test probes was determined. This variation was expected since there was no conductive scrim layer beneath the surface, but magnitude of the resistance with increasing distance was questioned. It was a pleasant surprise to discover that the slope of the line was very linear and *gradually* increased. It is interesting that extrapolating line out to 48" indicates that Rtt (calculated to be 1.54×10^7 ohms) is still well within the upper limits (1.0×10^9 ohms) of the requirements. ESD Workstations as defined in the Mil Spec are 48" wide. In addition, the slope was nearly the same at 10% relative humidity as compared to 50% relative humidity (3.0×10^5 and 1.9×10^5 respectively). This indicates that the resistance was relatively independent of humidity, which is favorable. Please compare figure 2 on page 2 with that of figure 5 on page 4. This data was taken after 48 to 68 hours of sample conditioning in the test environment.

A second resistance type of test was performed on sample 14 to determine the uniformity of resistance at a dozen independent points on the surface (Figures 3 and 6). This test used a 2.5 inch diameter probe consisting of an outside ring (guard, negative) and a positively charge disk about 1 inch in diameter at the center of the ring. This test is called Resitivity rather than Resistance and its units are in Ohms / Square rather than in Ohms. This test requires a really flat surface to get accurate results. The data in the tables adjacent to figures 3 and 6 has to be multiplied by 10 to get Ohms/Square. This was only done for the average values indicated beneath the tables. The values at the different points are pretty consistent, which is a good indication of surface uniformity.

Lastly, the important Static Dissipation test was performed on all samples at both 10% and 50% relative humidity. Since sample 14 was larger, it was possible to reposition it so the charged plate could contact the surface at different points. The samples were grounded during this test procedure at two different points. Alligator clips were attached to two of the hold down bolts on sample 14; whereas, two of the corners of samples 1 and 2 were grounded via the five-pound conductive weights. The test requirement calls for three 24 x 24" samples, but this is little hard to obtain with laboratory fabrication. Static Dissipation of all three samples was very good at both 10% and 50% RH, refer to pages 3 and 5. Between + and – 200 Volts is considered passing, and these samples were between + and – 50 volts. The amount of variation due to procedural errors in this test has not been reported in the literature.

Summary

Sample 14 was evaluated for Rtt, Uniformity, and Static Dissipation at 10 and 50% relative humidity and found to be satisfactory. Hardness testing was not performed. Samples 1 and 2 appear to be adequate but were not large enough for thorough testing.

Note:

Since conductive material was placed only in the top layer of paper, this experiment pretty conclusively shows the importance of putting the conductive material in that layer. Past efforts by Melamine Hard Laminate Manufacturers have focused on putting the conductive material beneath a top layer of pure melamine, which is an insulator. However, this is not saying that only the top layer should have conductive properties.

Prepared by AF Research Labs, Materials Directorate

STATIC DISSIPATIVE WORK SURFACES
SAMPLE # 3

Manufacturer:	Name:	METSS
	Address:	720 Lakeview Plaza Blvd Columbus, OH 43085
	P.O.C.	Dr. Ken Heater
	Phone:	614-842-6600
	FAX:	
Markings:	P/N or Other:	Sample #14
	Type of Surface:	Melamine Hard Laminate
	Subsurface:	Phenolic
	Conductive Polymer:	
	Scrim Layer:	None
	Adhesive:	None
Physical:	Weight:	
	Length:	18 1/4 "
	Width:	16 1/8"
	Thickness:	
	Number Layers:	Unknown
	Color:	Navy Blue, Textured
	Other:	Semi Gloss
	Mounting:	The laminate was mounted to 3/4" furniture grade plywood using 8 bolts to hold it flat.

Note: Samples 1 and 2 were also given limited testing. They were 6" square. Refer to Memo from K. Heater titled "Phase 1 Technical Feasibility Demonstration" Page 5 for better sample descriptions.

Special Features: Thin, slight warping

Test Equipment used in the following analysis:	Meg Ohm Meter:	Hewlett Packard 4339B High resistance Meter. The voltage had to be cut back to protect the meter from over current. The applied voltage is indicated in the accompanying data.
	Probes:	5 LB, Conductive Rubber faced
	Charge Dissipation:	This device is described in Appendix C of MIL-PRF-87893B
	Environmental Chamber:	Thermotron, calibrated annually

Prepared by AF Research Labs, Materials Directorate

ANALYSIS OF SAMPLES # 14, 1, 2
Static Dissipative Melamine Hard Laminate

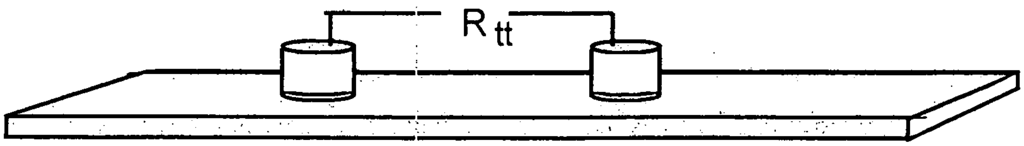


Fig. 1

Test Conditions: Date: ______ Temperature F: 72 Relative Humidity %: 10% Period of time (Hrs): 48

Sample # 14

Resistance Tests:
Top to Top (Rtt)

On the top side.

The acceptable range is:
1.00E+06 to 1.00E+09

Resistance	Distance "	DC Voltage
1.24E+06	4	100
1.71E+06	6	100
1.94E+06	8	100
2.18E+06	10	100
2.45E+06	12	100
2.70E+06	14	100
3.18E+06	16	100
2.20E+06		

Average **Ohms** **2.20E+06**

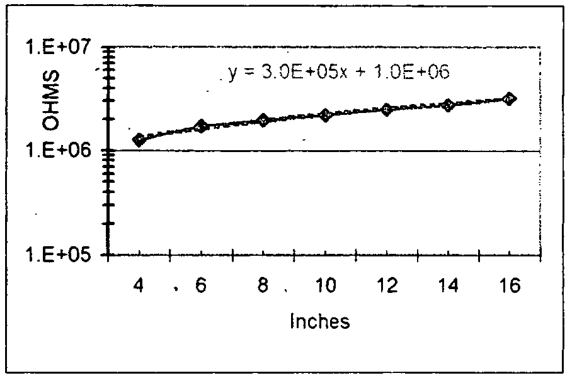


Fig. 2

Comments: This test passed.

The upward slope of this curve is typical of laminates without a scrim layer. If X = 48 " Y would = **1.54E+07** . This is not a very steep curve which is good.

Sample # 14

Resistivity Test:
10 Vdc at 15 sec.

Outer probe ring: **Negative**
Readings are in OHMS

Position	Reading	Position	Reading
1	1.96E+06	7	1.20E+06
2	5.00E+05	8	3.30E+05
3	9.00E+05	9	1.20E+06
4	1.10E+06	10	1.20E+06
5	5.40E+05	11	4.00E+05
6	1.00E+05	12	7.30E+05

Average **Ohms/Square** = 8.47E+05 x 10 = **8.47E+06**

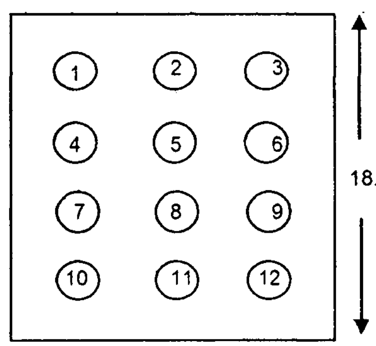


Fig. 3

Comments: This test is not required per Mil-PRF-87893B. It was used only as an indication of resistance uniformity since it measures over a small foot print.

The uniformity looks good.

Prepared by AF Research Labs, Materials Directorate

ANALYSIS OF SAMPLES # 14, 1, 2
Static Dissipative Melamine Hard Laminate

Mil-PRF-87893B Charge Dissipation Test

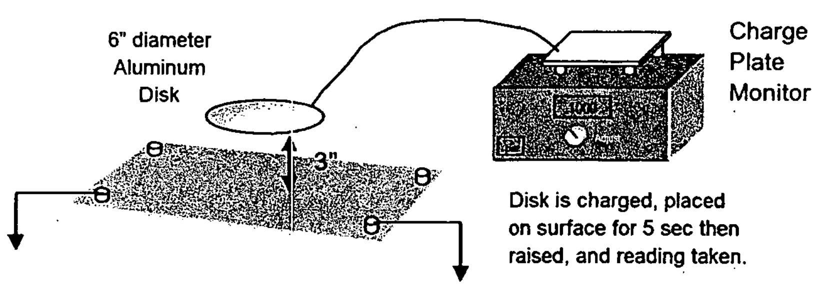


Fig. 4

Test Conditions: Date: ______ Relative Humidity %: 10%
Temperature F: 72 Period of time (Hrs): 48

Charge Dissipation: Appendix C of MIL-PRF-87893B
Acceptable value: Between + and - 200 Volts

Sample # 14

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	0	10	1	-2	-36
2	2	39	2	-1	4
3	1	8	3	-2	-12
4	2	17	4	-2	1

Sample # 1

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	0	12	1	-1	10
2	2	34	2	-1	8
3	1	12	3	-2	8
4	1	32	4	-3	8

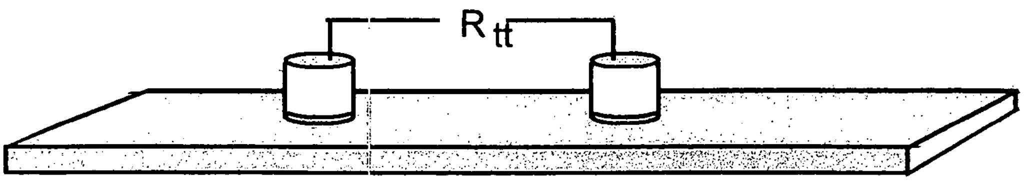
Sample # 2

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	0	11	1	-2	4
2	0	15	2	-1	9
3	0	13	3	-2	10
4	0	12	4	-1	11

Comments: All of the samples passed this test at 10% Relative Humidity.

Prepared by AF Research Labs, Materials Directorate

ANALYSIS OF SAMPLES # 14, 1, 2
Static Dissipative Melamine Hard Laminate



Test Conditions:	Date:		Relative Humidity %:	50 +/- 4%
	Temperature F:	72	Period of time (Hrs):	68

Sample # 14
Resistance Tests:
Top to Top (Rtt)
On the top side.

The acceptable range is:
1.00E+06 to 1.00E+09

Resistance	Distance "	DC Voltage
1.10E+06	4	100
1.30E+06	6	100
1.50E+06	8	100
1.70E+06	10	100
1.80E+06	12	100
2.00E+06	14	100
2.30E+06	16	100
1.67E+06		

Average **Ohms** **1.67E+06**

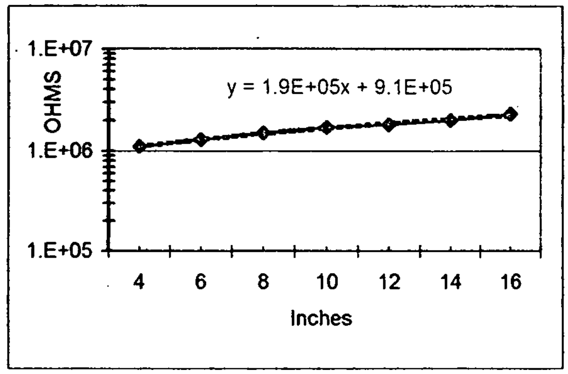


Fig. 5

Comments: This test passed. The upward slope of this curve is typical of laminates w/o a scrim layer. If X = 48", Y would = **1.00E+07** ohms. This curve is somewhat less steep than at 10% RH.

Sample # 14
Resistivity Test:
10 Vdc at 15 sec.
Outer probe ring:
Negative
Readings are
in OHMS

Position	Reading	Position	Reading
1	9.10E+05	7	9.20E+05
2	9.10E+05	8	2.30E+05
3	1.50E+06	9	9.10E+05
4	9.60E+05	10	9.70E+05
5	3.60E+05	11	2.50E+05
6	5.20E+05	12	7.80E+05

Average **Ohms/Square** = 7.68E+05 x 10 = **7.68E+06**

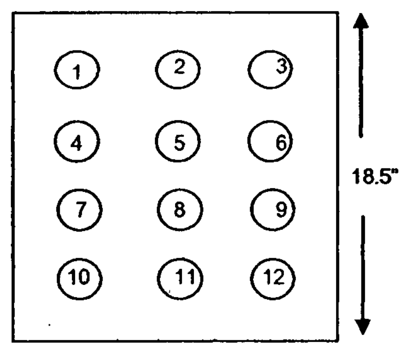


Fig. 6

Comments: The uniformity looks good.

Sample # 1 and 2
Resistivity Test:
100 Vdc at 15 sec.

		Outer Ring: **Negative**
Probe positioned	# 1	7.40E+06
at sample center.	# 2	5.70E+06
Average **Ohms/Square** =		**6.55E+07**

Resistance Tests:			Rtt
# 1	4"	2.10E+07	2.30E+07
# 2	4"	3.40E+07	1.80E+07
Avg **Ohms**		**2.75E+07**	**2.05E+07**

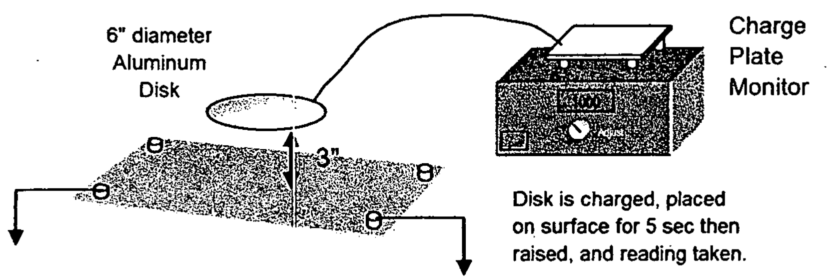
Comments: These tests passed.

These are prelininary resullts and do not infer product approval or endorsement. This data is not to be used in promotional literature.

Prepared by AF Research Labs, Materials Directorate

ANALYSIS OF SAMPLES # 14, 1, 2
Static Dissipative Melamine Hard Laminate

Mil-PRF-87893B Charge Dissipation Test



Test Conditions: Date: ______ Relative Humidity %: 50 +/- 4%
Temperature F: 72 Period of time (Hrs): 68

Charge Dissipation: Appendix C of MIL-PRF-87893B
Acceptable value: Between + and - 200 Volts

Sample # 14

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	3	43	1	0	2
2	3	38	2	-2	1
3	3	47	3	-3	0
4	0	3	4	-2	-6

Sample # 1

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	2	13	1	-1	9
2	2	14	2	-1	9
3	2	14	3	-1	9
4	2	15	4	-1	7

Sample # 2

+1000 Volts Applied			-1000 Volts Applied		
Trial	Dwn Volts	Up Volts	Trial	Dwn Volts	Up Volts
1	0	10	1	-1	5
2	0	3	2	-1	8
3	0	3	3	-1	7
4	0	0	4	-1	9

Comments: All of the samples passed this test at 50% Relative Humidity.

CONFIDENTIAL

Invention Disclosure

DATE:

TITLE: ESD Hard Laminate Surfaces

INVENTORS:

K. Heater, K. Pollard, M. Hodge, G. Salee

DATE OF FIRST ACTUAL OR ANTICIPATED PUBLIC DISCLOSURE:

unknown

SUMMARY OF THE INVENTION:

Current materials used for static discharge applications in hard laminates (e.g., work surfaces, flooring) do not provide the required levels of charge dissipation under conditions of low relative humidity required for today's advanced electronics, creating a need for new ESD surfaces that enable static charge to be safely and effectively dissipated from equipment and components placed in contact with the surface, thereby affording ESD protection to equipment and personnel. New technologies for conductive polymers and ESD applications have tremendous potential to address a new set of high performance criteria brought about through recent advances in microelectronics processing. Recent advances in the development of inherently conducting polymer (ICP) technologies have precipitated in the availability of several commercially available ICP products that can be blended or otherwise incorporated into polymer matrix materials to create homogeneously conductive materials.

The current invnetion consists of a blend of a commercially available intrinsically conductive polymer (Bayer Baytron® P) with a standard melamine formaldehyde resin used in the production of commercial laminates (work surfaces, flooring, etc.). The formulations are stable and 100% water soluble (no VOCs). Solution concentrations up to 10% ICP have been formulated, but test results indicate that less than 1% ICP content may be adequate to address the ESD requirements set forth by the Air Force (MIL-PRF-87893B) including the following:

- *Point-to-Point Resistance* - Resistance values in the range of 1×10^6 to 1×10^9 ohms, measured using two probes placed 4 inches apart on center and recorded after 15 seconds of contact and a charge level of 100V.
- *Electrostatic Dissipation* - The remaining charge on a charge plate is less than 200V, with an initial charge of 1000V and determined after removing the plate after 5 seconds of contact with the ESD surface (i.e., performed in accordance with MIL-PRF-87893B).
- *Low Humidity Performance* - Samples demonstrate acceptable performance behavior at all relative humidty levels between 10 and 50%.

The chemistry of the system should essentially allow METSS to replace the current curing agent (Part B) used by Formica to cure their melamine resins with a new Part B that contains the ICP additive. The METSS ICP-B is an aqueous solution that mixes readily with the melamine resin system and has the right pH to effect cure in the same manner as the existing Part B curative. Thus, with the exception of

switching to a new Part B curative (METSS ICP-B), production methods for the ESD laminate systems should be unchanged.

WHEN DID YOU MAKE THE FIRST WRITTEN DESCRIPTION OF YOUR IDEA AND WHERE IS THIS WRITTEN DESCRIPTION?

Lab book records - K Pollard, Gordon Jones. Detailed in memo to Air Force on

HAVE YOU MADE ANY DRAWINGS OR SKETCHES OF YOU IDEA? IF SO, WHAT WAS THE DATE OF THE DRAWING AND WHERE IS IT?

No. Just written descriptions and data plots.

HAVE YOU TESTED YOUR IDEA ON AN EXPERIMENTAL BASIS? IF SO WHEN AND WHERE WAS THIS DONE AND WHAT WERE THE RESULTS?

Yes. In-house testing documented in Air Force memo. Air Force also did testing and documented results in memo to METSS on .

DESCRIBE HOW DISCLOSED:

Confidential Memos, progress reports and Final Report to Government with distribution limited to Government use.

DESCRIBE THE PRIOR ART:

Prior art uses salts (humectants) and conductive additives that are incorporated in the matrix of the laminate (through the thickness of the laminate). These systems do not meet performance requirements. Additional information attached.

LIST A MAXIMUM OF 8 KEY WORDS THAT DESCRIPE THE INVENTION:

conductive additive, antistatic, ESD, inherently conducting polymer, melamine, laminate

DETAILED DESCRIPTION OF INVENTION:

See attached memos and reports.

SIGNED AND WITNESSED:

Signed:

Witnessed:

MONTHLY STATUS REPORT

Contract No.: F33615-99-C-5606
SBIR Program: AF99-146
Program Title: Development of Static Dissipation Hard Laminate Surfaces
P.I.: Kenneth J. Heater
COR: Julius Brodbeck
Reporting Period:
Report No: 0001AA -

Project Objective

The project objective is to develop cost-effective static dissipation hard laminate surfaces which will pass MIL-PRF-87893B performance specifications for a Type I Rigid Work surface.

Project Status

Start of Work Meeting

A start of work meeting was held between METSS staff and Air Force personnel on Discussions included the following:

Proposal Review. The program plan submitted by METSS was reviewed. Several approaches were proposed by METSS to address the ESD issue:

1. use of proper additives (nanophase)
2. polymer blending
3. use or synthesis of intrinsically conducting polymers or co-polymers

The first two methods are preferred as they can be readily integrated into existing manufacturing processes (direct replacement - additional solid or liquid additive). Nanophase additives can presumably be used at higher loading levels without affecting mechanical properties and may not be as readily depleted from peaks in contact surface area, thereby creating a more homogeneous ESD system.

Commercialization. The importance of the commercialization aspects of the SBIR program was emphasized by the Air Force. METSS reviewed its general approach to product commercialization and outlined potential commercialization vehicles for the ESD program that included the potential for METSS to supply ESD resins or additives to ESD laminate manufacturers or licensing of the technologies developed under the SBIR program. Emphasis was placed on identifying a viable partner to team with METSS to support ESD laminate development and (ultimately) manufacturing efforts. The Air Force suggested potential companies of interest might include Formica and Spaulding Composites. Demonstration of technical capabilities and commercialization potential within the first 6 months of the

program was emphasized. The patent potential for ESD laminate systems based on nanophase additives was discussed.

Technical Discussion. METSS provided a technical overview of the proposed SBIR program efforts that included a review of the three approaches proposed by METSS and the advantages and disadvantages of each approach. The Air Force reviewed the technical requirements of program, which included a review of the ESD laminate use, desired property characteristics, and testing and qualification efforts. The Air Force volunteered to test the best candidate surfaces developed by METSS and provide METSS with direction needed to support the in-house testing and evaluation efforts desired by METSS to direct formulation development efforts. The design of the current laminate structure was reviewed along with short and long term design change objectives. The Air Force suggested a portable mat with rubber backing would be good prototype target for the SBIR program effort.

Technical Efforts

Technical efforts performed during the first phase of the program efforts emphasized the following:

- Gathering and reviewing technical information needed to support the program efforts. These efforts have been support by searches of technical databases, the patent literature, and commercial product information. Pertinent literature has been reviewed and used as a basis to direct the initial program development efforts. Emphasis is being placed on identifying nanophase technologies capable of supporting the initial program development efforts.
- Becoming familiar with commercial laminate production methods. See commercialization.
- Ordering materials needed to support the program development efforts. METSS identified a number of commercial companies capable of providing nanophase additives that may be sued to support the ESD laminate development efforts. METSS has contacted these companies and ordered sample products to support our formulation development effort. METSS also ordered melamine resin that is consistent with the product currently used by Formica in their commercial production process. Formica also provided paper substrate materials for use in the laminate constructions.
- Developing capability to support in-house ESD testing efforts. METSS contacted a number of companies that provide the type of charge plate monitors needed to support the in-house ESD testing efforts. Purchase price, lease price, and performance information was obtained. The Air Force is being consulted for direction in this regard. METSS is also looking at acquiring or constructing a glove box to facilitate humidity control for ESD testing efforts.
- Performing baseline formulation development efforts. METSS has initiated formulation development efforts with additives that have been received to date. Efforts are currently focused on the development of high quality, homogenous laminates. Performance issues will be addressed once good quality systems are produced.
- Developing production methods for lab-scale samples. METSS has developed capability to use its lab press to make suitable samples to support the Phase I program efforts. Production methods are being optimized (i.e., time, temperature, pressure, loading).

Commercialization Efforts

METSS and Air Force personnel visited the Formica plant in Cincinnati, OH in June to discuss possible commercialization strategies and to observe industrial laminate manufacturing techniques.

A brief meeting was held at the beginning of the facility visit, during which METSS outlined the technical goals and strategies associated with the Phase I program, along with possible strategies for commercialization of the technology developed under the Phase I program, during a more focused Phase II development. The meeting opened lines of communication between METSS and Formica, and Formica has agreed to provide program support to METSS throughout the Phase I program and beyond. At present, Formica has no commercial ESD laminate line, and has indicated an eagerness to work with METSS to incorporate METSS' proposed technology into a commercially available ESD laminate system. Initially, Formica will provide in-kind support in the form of formulation assistance, material supply and testing capabilities. It is anticipated that Formica will also provide assistance with scale-up and prototype manufacture in a pilot plant at their facility in Cincinnati.

The tour of the plant has provided METSS with a clear understanding of process variables and practices relevant to fabrication and testing of laminate materials, which will ensure that program development efforts proceed along the most industrially relevant lines possible. Additionally, knowledge of the process and the ability to work closely with Formica as a potential commercialization partner should facilitate a smooth transition from lab scale fabrication to commercial scale production as part of a Phase II program.

Since the plant visit, METSS has initiated follow-up discussions with Formica on the development of a business relationship between the two companies for the purposes of developing a commercially available ESD laminate product.

Anticipated Progress (Next Reporting Period)

- METSS will continue to develop technical concepts that may support the program objectives based on information derived from the literature.
- Continue identifying and acquiring materials needed to support initial Phase I efforts.
- Determine coating procedures for nano-particulate materials that may be required to support the Phase I program and develop a plan of implementation.
- Develop appropriate analysis techniques needed to support the Phase I program efforts.
- METSS will design and perform screening experiments to determine the efficacy of various approaches to designing the ESD laminate. This effort will include the evaluation of nano-particles and/or conductive polymeric additives.

<u>Phase I Work Schedule</u>

Projected vs. Actual Performance

Task Description	Month								
	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
Task 1. Start of Work Meeting									
Progress	♦								
Task 2. Technical Review									
Progress									
Task 3. Processing & Fabrication Development									
Progress									
Task 4. Testing and Evaluation									
Progress									
Task 5. Prototype Fabrication and Testing									
Progress									
Task 6. Administrative & Reporting			M		M		M		M F

M = Monthly Progress Report; F = Final Report

MONTHLY STATUS REPORT

Contract No.: F33615-99-C-5606
SBIR Program: AF99-146
Program Title: Development of Static Dissipation Hard Laminate Surfaces
P.I.: Kenneth J. Heater
COR: Julius Brodbeck
Reporting Period:
Report No.: 0003AA –

Project Objective

The project objective is to develop cost-effective static dissipation hard laminate surfaces that will pass MIL-PRF-87893B performance specifications for a Type I Rigid Work surface.

Project Status

Manufacturing Development Efforts. The ability to produce high quality laminates is critical to the success of the program as surface irregularities and imperfections can affect ESD performance. As such, efforts were undertaken to develop the capabilities needed to support prototype laminate production efforts. Initial efforts focused on the production of conventional (non-ESD) laminates. Through on-going discussions with Mr. Terry Drees of Formica, METSS has become familiar with Formica's commercial laminate production methods and the materials used in the construction of high quality commercial laminates. This information has been used to direct our prototype development efforts in a way that will ensure the samples produced for testing and characterization under the SBIR program provide a realistic expectation of performance properties that may be obtained using commercial production methods. In addition to providing technical support, Formica has provided materials to support the prototype development efforts including paper for the clear coat, and a variety of resin impregnated substrate papers for use in the laminate constructions. METSS has also ordered melamine resin that is consistent with the product currently used by Formica (provided by Capital Resins Corporation, the supplier of Formica's commercial production resins) and receives 'fresh' batches of resin every second week as the melamine has a tendency to precipitate out of solution after a period of time. Key issues encountered and addressed during the production of viable prototype samples included the identification of proper processing conditions (i.e., temperature, pressure and time), the determination of proper drying times for the resin impregnated sheets to ensure laminate quality, issues associated with the creation of a flat laminate suitable for ESD testing, the overall consistency and surface quality of the end laminate, and problems associated with the laminate adhering to the press plates. Each of these issues was addressed in time and METSS is currently producing high quality 6" x 6" prototype laminates on a consistent basis. However, as the properties of the resin have a significant effect on processability, these efforts will have to be refined for each series of ESD formulations developed under the program. ESD samples that have been prepared by METSS based on the ESD formulation development efforts performed during this reporting period and described subsequently in this proposal are described in Table 3.

ESD Testing Development Efforts. In-house ESD testing capabilities are necessary to support the formulation development efforts in the most efficient manner possible. METSS' approach in this regard has not been to attempt to recreate the Air Force testing capabilities, but rather to develop enough in-house capability to evaluate the ESD samples being developed by METSS to direct our formulation design efforts. ESD samples demonstrating the required performance during in-house testing will be submitted to the Air Force for accurate characterization. In support of this effort, METSS contacted a number of companies that provide charge plate monitors to obtain technical specifications and pricing information. After this information was reviewed with the Air Force program monitor, METSS purchased a charge plate monitor to support the in-house sample testing and evaluation efforts. In the absence of adequate in-house capabilities to control relative humidity and temperature as provided in the MIL-spec, METSS will perform ESD measurements in a dedicated controlled-atmosphere glove box, constructed to support the ESD test measurements. The glove box will provide a constant humidity environment required to support the ESD characterization efforts by using standard aqueous salt solutions to obtain the desired humidity levels. All experiments in METSS' in house testing efforts will be performed at room temperature. A temperature/relative humidity sensor will be used to monitor exact conditions during ESD testing. Two charge plate probes, a standard 6-inch diameter probe and a smaller 4-inch diameter probe (test samples are 6-inch squares, so edge effects were of concern), were fabricated in accordance with the MIL-specs. A pulley system was constructed to lower and raise the charge plate at a controlled rate, and METSS is currently constructing a mechanized timing/motor system to support full automation of the ESD test. The Air Force is providing control samples to support the program efforts. METSS will record measurements made on at least one of these control samples prior to every measurement performed on a METSS ESD laminate so that the data can be normalized accordingly.

A meeting held on the 1st of September with the Air Force program monitor encompassed a review of the test apparatus set-up and ESD measurement techniques. It was demonstrated that METSS could conduct screening tests outside of the glove box as RH is currently about 35-40%. Testing appears to be very straight forward, so METSS should be able to produce good in-house results to support our formulation development efforts. METSS will have to use a desiccant inside of the glove box to support testing at reduced humidity levels.

Formulation Development Efforts. The program plan submitted by METSS included several approaches to address the ESD issue including the use of proper additives (nanophase), conductive polymer blending, and the synthesis of intrinsically conducting polymers or co-polymers. The first two methods were emphasized as they can be readily integrated into existing manufacturing processes. While METSS has begun to look at approaches that encompass conductive polymer blending techniques, most of the efforts expended by METSS during the reporting period were associated with the development of ESD laminates based on nanophase additives.

At the onset of the program METSS staff carried out a number of experiments to support baseline formulation development efforts using higher loading levels of nanophase additives to obtain a quick idea of the types of problems that will be encountered under the program. Initial experiments using nanoparticles in a model resin system (polyvinyl alcohol) were performed to assess the general dispersion characteristics and the effects of loading levels on resin properties. Sample testing and evaluation efforts used to support the initial ESD formulation development efforts included visual inspection methods, optical microscopy, SEM, and contact probe conductivity measurements. These experiments, and subsequent experiments performed in the melamine resin system quickly demonstrated that dispersion problems were going to be a significant challenge and that proper dispersion methods were going to have to be developed for each conductive nanophase additive to address both ESD and physical property requirements. As outlined further in this report, more sophisticated methods were

required to support the development of techniques for proper dispersion of the nanophase additives including particle size analysis techniques and isoelectric point measurements.

Particle size analysis measurements were performed using CAPA 500 Centrifugal Automatic Particle Analyzer. The CAPA 500 gives information on particle size distributions based on Stoke's equation calculations. Since it determines the number of particles in a certain size range and reports that data with an average particle size at the end of the test, the results are approximate within the size range and are best used in showing trends. However, the CAPA 500 does provide an effective method of evaluating dispersion techniques and optimizing nanophase dispersions.

Isoelectric point measurements were performed using a MATEC ESA 8000. Isoelectric point measurements are desired to determine the pH range required for proper dispersion of nanophase additives (pH is a critical issue in nanophase particle dispersion is solution). For most oxides, as pH is increased, the adsorption of potential determining ions, H^+ and OH^-, changes in correspondence with the concentration of these species in solution. For each surface, therefore, a point is reached at which the concentration of positive ions and negative ions is balanced, the point of zero charge, and is called the isoelectric point (determined by measuring the zeta potential over a range of pH values).[1] The zeta potential measurements give two important pieces of information: pH ranges over which a particle is stable to dispersion (pH values away from the isoelectric point) and the magnitude of the zeta potential gives an indication of the need for dispersants to obtain a stable dispersion.

Current ESD formulation development efforts are being supported by the use of the following nanophase additives:

- $SbSnO_2$ - a light blue/grey powder with average particle size of 90 nm and density 6.43 g/mL. It is manufactured by Nanophase Technologies, Burr Ridge, Il.
- Printex L6 - a carbon black with an average particle size of 18 nm and a density of 350g/L. It is manufactured by Degussa Rubber Chemicals and Pigments, Akron, OH.
- Printex L - a carbon black with an average particle size of 23 nm and a density of 400g/L. It is manufactured by Degussa Rubber Chemicals and Pigments, Akron, OH.
- Carbon black nanotubes - carbon fibers with a density of 2.0 g/mL. It is manufactured by Hyperion Catalysis International, Cambridge, MA.

METSS is awaiting arrival of the following specialty nanophase additives from Micronisers:

- fluorine doped ZnO
- doped SnO
- other naonphase carbon.

These additives are being provided in bulk form and as specially treated additives to support melamine incorporation.

In addition to obtaining nanophase additives from commercial and specialty suppliers to support the ESD development efforts, METSS has also investigated the potential of creating coated nanophase clays as a possible nanophase component to conductive resins. Preliminary studies indicate that it is possible to coat montmorillonite clay with silver by use of a simple oxidation-reduction reaction to achieve a conductive

[1] Terry A. Ring, "Fundamentals of Ceramic Powder Porcessing and Synthesis." Academic Press, San Diego, Chapter 9, 1996.

sample. Further work in characterizing the coated clay and in characterizing resins with clay incorporation has indicated that, since silver is a light sensitive metal, on continued exposure to light, it decomposes and forms colloidal silver, changing color from off-white to a brown/purple. As a result, silver coatings were eliminated. Copper is still being investigated as alternate route. However, coating of the montmorillonite clay with copper is more difficult than coating the clay with silver.

During the current reporting period METSS also started to pursue efforts related to the development of ESD laminates based on conductive polymer blends. These efforts are being supported by the use of commercially available materials. METSS identified several candidate technologies for evaluation under the program and, to date, has obtained one sample of a conductive resin additive for testing and evaluation. This sample has been provided by Bayer under the trade name Baytron P. Baytron P is a water soluble additive based on polythiophene chemistry that can be readily blended with melamine resin to form a durable crosslinked system. Conductive blends based on this material, or other like it, may provide a viable route to obtaining the program objectives by themselves or when used in conjunction with nanophase additive systems. As noted in Table 3, sample color may be one of the problems encountered with the use of conductive resins. Other conductive resin technologies currently under consideration by METSS include a polyphenyleneamine polymeric salt provided by Ormecon of Ammersbek, Germany, and conductive polymer technologies developed by Mearthane Products Corporation, Cranston, R.I.

While the Baytron P material has been fairly easy to work with, the primary challenge associated with the use of nanophase additives is *dispersion*. A discussed in our original proposal, this is a significant problem that often requires a substantial amount of trial and error to address. METSS performed some trial and error experiments to get a feel for the types of problems that we were going to encounter under the program. However, a number of tasks were also performed to develop methodologies required to address the issue of nanophase dispersion on a more fundamental level. A brief overview of some of the formulation development efforts is provided.

Several methods of nanophase dispersion were evaluated during the reporting period including combinations and permutations of high shear dispersion methods and ultrasonic techniques. High sheer methods for nanophase dispersion were evaluated first, but with limited success (Table 1). Samples for particle size analysis were taken from a sheared solution of nanoparticles in water and a sheared solution in melamine formaldehyde resin in water (MF = 60%MF and 40% H_2O) and tested using the CAPA 500 to determine the effectiveness of shearing on particle deagglomeration. The results of the tests performed by METSS demonstrated that high shearing rates and longer shearing times did not provide for adequate particle deagglomeration.

Table 1. Effect of Shear Time on the Average $SbSnO_2$ Particle Size

Amt. of $SbSnO_2$ (g)	Liquid Phase (g)	Shear time (min.)	Ave. particle size (μm)
2.5	47.5 H_20	20	1.15
2.5	47.5 H_20	30	0.52
2.5	47.5 H_20	40	0.86
1.58	50 MF	20	1.50
1.58	50 MF	30	1.53
1.58	50 MF	40	1.72

The use of ultrasonic techniques in conjunction with proper control of solution characteristics appear to be the key to obtaining proper dispersion of the nanophase additives. Ultrasonics are used often in powder deagglomeration processes where other dispersion techniques fail. In theory, ultrasonic techniques work because of local cavities that are formed during ultrasonication where very high concentrations of high velocity jets in the neighborhood of 100 m/s and pressure gradients of 20 GPs/cm are formed.[1] The resulting mechanical forces on aggregated particles in the vicinity of the cavity are extremely strong – strong enough to break apart weakly bonded particles (for example, Van Der Waals forces). In an effort to evaluate the use of ultrasonic techniques in the present application, METSS carried out a series of tests on sonicated solutions of $SbSnO_2$ in water (using a sonic bath and a sonic horn), removing an aliquot of the solution every 10 minutes over a time frame of about an hour, to determine optimum conditions for particle deagglomeration. The aliquots were tested using the CAPA 500 particle analyzer and the results are shown in Table 2. The fundamental description of the effects of ultrasonication on dispersed agglomerate particles is extremely complex and depends on both the probability of deagglomeration and of agglomeration.[1] This effect is seen in the results of Table 2. After 40 minutes of sonication, the dispersed particles are beginning to interact more often with themselves, causing reagglomeration (seen as an increase is average particle size), instead of interacting with cavities created by ultrasonication and becoming deagglomerated. The results generated with the ultrasonic horn were deemed to be the most favorable as sonicating the 10 wt% $SbSnO_2$ solution for 40 minutes produced optimum agglomeration break up. Equivalent results were not obtained with the sonic bath even after 2 hours of sonication.

Table 2. Effect of Sonicating on $SbSnO_2$ Particle Size in an Aqueous Solution

Sonication Time (min.)	Ave. Particle size (nm) – Ultrasonic Bath	Ave. Particle size (nm) – Ultrasonic Horn
20	210	140
30	210	110
40	200	90
50	190	100
60	170	100
70	170	120

METSS was pleased with the results of the sonicating experiments with regards to the $SbSnO_2$ nanophase particles and is confident that the use of ultrasonic techniques will make it possible to address particle dispersion issues in an effective manner. METSS is currently testing some of its other nanophase additives to determine their behavior under similar conditions.

As previously mentioned, the ability to properly disperse nanophase additives in a solution is strongly dependent on solution pH. To begin addressing this issue, METSS determined the isoelectric point for the $SbSnO_2$ nanoparticles. From the results of our studies, METSS learned that the isoelectric point of $SbSnO_2$ is very low, and as a result $SbSnO_2$ can be dispersed over a wide pH range. In addition, the magnitude of the zeta potential (-40 mV) is large and indicates that a dispersing agent should not be necessary to stabilize the dispersion. From a practical standpoint, this result implies that it will not be difficult to disperse the $SbSnO_2$ nanoparticles in the melamine resin system at any practical pH level.

METSS is currently testing other nanophase particles to learn more about their properties to support the dispersion of these materials. Preliminary isoelectric point experiments were performed on carbon black nanoparticulate materials obtained to support the ESD program development efforts, including dispersions of two different grades of carbon black. The more highly conducting sample (XE2) had a very large surface area which led to high solvent absorption. As such, a well-dispersed solution could not be obtained in appropriate concentrations to support the test. A second carbon black (L) was also prepared for analysis. Dispersions were difficult but a solution of appropriate concentration was obtained in very acidic conditions ($pH \approx 2$). Tests of the isoelectric point were, however, ineffective due to the particle properties in solution. The only conclusions from theses test were that carbon black dispersions in very acidic solutions were possible. Using this information, METSS carried out a series of particle deagglomeration experiments on a carbon black sample at pH = 2, using the sonic bath and testing the particle breakup using the CAPA 500. However, at the time of this report, sample handling difficulties have generated unreliable data.

Table 3. Formulations of Preliminary Laminates

Nanophase Material	pH	Weight Percent		Volume Percent		Final Appearance
		Particle	Resin	Particle	Resin	
$SbSnO_2$	2.85	5	95	0.6	99.4	Hazy blue
$SbSnO_2$	11.6	5	95	0.06	99.4	Hazy blue
C black (L)	0	0.2	99.8	0.6	99.4	Black/Grey
C black (L)	0	0.08	99.92	0.16	99.84	Hazy Grey
C black (L)*	2	0.08	99.92	0.16	99.84	
C black (L6)		0.18	99.82	0.6	99.4	
C fibers	2	1.6	98.4	0.6	99.4	
Baytron		5	95			Blue

- The melamine formaldehyde resin density is $\rho = 1.215\text{-}1.235\ g/cm^3$ depending on the batch.
- $\rho(SbSnO_2) = 6.43 g/cm^3$, ρ(C black- L) = $0.400 g/cm^3$, ρ(C black- L6) = $0.350 g/cm^3$, ρ(C fibres) = $2.0 g/cm^3$.

* Surfynol surfactant in ethylene glycol was added at 0.5 wt % of total mixture weight.

Initial ESD Test Results

The results of some quick testing performed by the Air Force program monitor on some of the ESD laminate samples prepared by METSS to date were promising. The Baytron sample was clearly the best performer, demonstrating proper conductivity and passing the ESD test at 37%RH. In addition, one of the 5% $SbSnO_2$ samples tested very well, demonstrating proper conductivity, but not quite passing the ESD test. The results of the testing performed on the available samples clearly indicated that processing of the samples containing the nanophase additives will be extremely important for ESD performance.

Anticipated Progress (Next Reporting Period)

- METSS will continue to develop technical concepts that may support the program objectives based on information derived from the literature.
- Continue identifying and acquiring materials needed to support initial Phase I efforts.

- Complete the set up and familiarization procedures required for effective use of the charge plate monitor and the humidity chamber.
- Analyze data generated from the ESD tests and use it to reformulate any step in our laminate procedure that needs to be adjusted to reach our goals.
- Determine coating procedures for nano-particulate materials that may be required to support the Phase I program and develop a plan of implementation.
- Use appropriate analysis techniques needed to support the Phase I program efforts, (for example SEM, TEM).
- METSS will design and perform screening experiments to determine the efficacy of various approaches to designing the ESD laminate. This effort will include the evaluation of nano-particles and/or conductive polymeric additives.
- METSS will use the isoelectric point determination data to direct the design of the ESD laminate. Each type of nanoparticle will have pH-dependent dispersion properties that may allow them to be incorporated into different parts of the laminate preparation. For instance, the dispersion of the carbon black nanophase particles (L) was only accomplished in very acidic solutions (pH = 2) and would not succeed in basic or neutral solutions. At the same time, the isoelectric point determination data can be used to indicate where there is room for negotiation. For instance, $SbSnO_2$ is acidic in solution and can be used as the curing agent for the laminates. Conversely, $SbSnO_2$ is also stable in basic solution and so can be dispersed within the melamine formaldehyde resin (pH ≈ 10) itself and then cured later. Understanding the properties of the nanophase particles that are used allows METSS more control over the outcome of the laminate.
- METSS will continue to accumulate and evaluate data to further advance the project.

Phase I Work Schedule

Projected vs. Actual Performance

Task Description	Month								
	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
Task 1. Start of Work Meeting									
Progress	♦								
Task 2. Technical Review									
Progress									
Task 3. Processing & Fabrication Development									
Progress									
Task 4. Testing and Evaluation									
Progress									
Task 5. Prototype Fabrication and Testing									
Progress									
Task 6. Administrative & Reporting			M		M		M		M F

M = Monthly Progress Report; F = Final Report

MONTHLY STATUS REPORT

Contract No.: F33615-99-C-5606
SBIR Program: AF99-146
Program Title: Development of Static Dissipation Hard Laminate Surfaces
P.I.: Kenneth J. Heater
COR: Julius Brodbeck
Reporting Period:
Report No.: 0004AA –

Project Objective

The project objective is to develop cost-effective static dissipation hard laminate surfaces that will pass MIL-PRF-87893B performance specifications for a Type I Rigid Work surface.

Project Status

Manufacturing Development Efforts. The ability to produce high quality laminates is critical to the success of the program as surface irregularities and imperfections can affect ESD performance. As such, efforts were undertaken to develop the capabilities needed to support prototype laminate production efforts. Through on-going discussions with Mr. Terry Drees of Formica, METSS has become familiar with Formica's commercial laminate production methods and the materials used in the construction of high quality commercial laminates. This information has been used to direct our prototype development efforts in a way that will ensure the samples produced for testing and characterization under the SBIR program provide a realistic expectation of performance properties that may be obtained using commercial production methods. In addition to providing technical support, Formica has provided materials to support the prototype development efforts including paper for the clear coat, a variety of resin impregnated substrate papers for use in the laminate constructions and the materials required to make several 18" x 18" laminate samples. Key issues encountered and addressed during the production of the 6" x 6" viable prototype samples included the identification of proper processing conditions (i.e., temperature, pressure and time), the determination of proper drying times for the resin impregnated sheets to ensure laminate quality, issues associated with the creation of a flat laminate suitable for ESD testing, the overall consistency and surface quality of the end laminate, and problems associated with the laminate adhering to the press plates. With samples produced on the small production line using a METSS additive (described in more detail later), the major issue was concentration of the melamine solution, i.e., the dilution factor. To use a diluted solution of melamine formaldehyde resin to coat a sheet of cellulose paper, the rate at which the paper is dipped into the resin solution must be slowed down in order to coat as much resin on the paper as would be deposited there if a more concentrated solution was used. Theoretically, slowing down the paper coating rate until enough resin is added to the paper should give a successful coating provided it is placed on evenly, however in reality limits are reached. The paper will eventually reach a saturation point, at which time the resin will begin to puddle on the sheet, leaving streaks in the final product. In addition, after a length of soaking, the paper strength decreases causing the paper to tear. These issues were addressed as they were encountered and METSS is currently producing high quality prototype laminates on a consistent basis.

ESD Testing Development Efforts. In-house ESD testing capabilities are necessary to support the formulation development efforts in the most efficient manner possible. METSS' approach in this regard has not been to attempt to recreate the Air Force testing capabilities, but rather to develop enough in-house capability to evaluate the ESD samples being developed by METSS to direct our formulation design efforts. Several ESD samples which demonstrated the required performance during in-house testing were submitted to the Air Force for further characterization. In the absence of adequate in-house capabilities to control relative humidity and temperature as provided in the MIL-spec, METSS has performed and will continue to perform ESD measurements in a dedicated controlled-atmosphere glove box, constructed to support the ESD test measurements. The glove box provides a constant humidity environment required to support the ESD characterization efforts by using standard aqueous salt solutions or desiccants and dry nitrogen gas to obtain the desired humidity levels. METSS has conducted all experiments at relative humidity levels of 20, 30, and 40% and all experiments in METSS' in house testing efforts have been performed at room temperature. A temperature/relative humidity sensor has been used to monitor exact conditions during ESD testing. Two charge plate probes, a standard 6-inch diameter probe and a smaller 4-inch diameter probe (test samples are 6-inch squares, so edge effects were of concern), were fabricated in accordance with the MIL-specs. A pulley system was constructed to lower and raise the charge plate at a controlled rate, and METSS is currently constructing a mechanized timing/motor system to support full automation of the ESD test. The Air Force provided control samples to support the program efforts. METSS has recorded measurements made on all three of these control samples prior to every measurement performed on a METSS ESD laminate so that the data could be normalized accordingly.

Formulation Development Efforts. The program plan submitted by METSS included several approaches to address the ESD issue including the use of proper additives (nanophase), conductive polymer blending, and the synthesis of intrinsically conducting polymers or co-polymers. The first two methods were emphasized as they can be readily integrated into existing manufacturing processes. METSS has developed approaches that encompass conductive polymer blending techniques and the development of ESD laminates based on nanophase additives.

Throughout Phase I, METSS has emphasized the need to obtain good and stable dispersions of the nanophase particles and has actively pursued methods to evaluate the dispersions. In this regard, particle size analysis measurements were performed using CAPA 500 Centrifugal Automatic Particle Analyzer. The CAPA 500 gives information on particle size distributions based on Stoke's equation calculations. Since it determines the number of particles in a certain size range and reports that data with an average particle size at the end of the test, the results are approximate within the size range and are best used in showing trends. However, the CAPA 500 does provide an effective method of evaluating dispersion techniques and optimizing nanophase dispersions.

Isoelectric point measurements were performed using a MATEC ESA 8000. Isoelectric point measurements are desired to determine the pH range required for proper dispersion of nanophase additives (pH is a critical issue in nanophase particle dispersion is solution). For most oxides, as pH is increased, the adsorption of potential determining ions, H^+ and OH^-, changes in correspondence with the concentration of these species in solution. For each surface, therefore, a point is reached at which the concentration of positive ions and negative ions is balanced, the point of zero charge, and is called the isoelectric point (determined by measuring the zeta potential over a range of pH values).[1] The zeta potential measurements give two important pieces of information: pH ranges over which a particle is

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[1] Terry A. Ring, "Fundamentals of Ceramic Powder Porcessing and Synthesis." Academic Press, San Diego, Chapter 9, 1996.

stable to dispersion (pH values away from the isoelectric point) and the magnitude of the zeta potential gives an indication of the need for dispersants to obtain a stable dispersion.

Current ESD formulation development efforts are being supported by the use of the following nanophase additives:

- $SbSnO_2$ - a light blue/grey powder with average particle size of 90 nm and density 6.43 g/mL. It is manufactured by Nanophase Technologies, Burr Ridge, Il.
- Printex L6 - a carbon black with an average particle size of 18 nm and a density of 350g/L. It is manufactured by Degussa Rubber Chemicals and Pigments, Akron, OH.
- Printex L - a carbon black with an average particle size of 23 nm and a density of 400g/L. It is manufactured by Degussa Rubber Chemicals and Pigments, Akron, OH.
- Carbon black nanotubes - carbon fibers with a density of 2.0 g/mL. It is manufactured by Hyperion Catalysis International, Cambridge, MA.
- fluorine doped ZnO – an off-white powder with density of about 5.6g/mL. There are two varieties with 1% and 2% dopant. It is manufactured by Micronisers of Melbourne, Australia.
- other nanophase carbon – It is manufactured by Micronisers of Melbourne Australia.

During the current reporting period METSS also pursued efforts related to the development of ESD laminates based on conductive polymer blends. These efforts are being supported by the use of commercially available materials. METSS identified several candidate technologies for evaluation under the program and, to date, has obtained two samples of conductive resin additives for testing and evaluation. One sample has been provided by Bayer under the trade name Baytron P. Baytron P is a water soluble additive based on polythiophene chemistry that can be readily blended with melamine resin to form a durable crosslinked system. Conductive blends based on this material, or other like it, may provide a viable route to obtaining the program objectives by themselves or when used in conjunction with nanophase additive systems. As noted in Table 2, sample color may be one of the problems encountered with the use of conductive resins. The second sample was provided by CIBA Chemicals under the trade name Irgastat P22. It is an insoluble additive based on nylon. To this point, METSS has not successfully incorporated it into a resin formulation. It is most commonly used in applications where it can be dispersed at its melt temperature (220°C). Application of this system to the melamine formaldehyde resin system is difficult since melamine formaldehyde resin is not stable in this temperature range. Other conductive resin technologies currently under consideration by METSS include a polyphenyleneamine polymeric salt provided by Ormecon of Ammersbek, Germany, and conductive polymer technologies developed by Mearthane Products Corporation, Cranston, R.I.

While the Baytron P material has been fairly easy to work with, the primary challenge associated with the use of nanophase additives is *dispersion*. As discussed in our original proposal, this is a significant problem that often requires a substantial amount of trial and error to address. A brief overview of some of the progress in formulation development efforts for this reporting period is provided.

The formulation section of the last bimonthly reporting period included characterization of the nanophase $SbSnO_2$ and development of good methods for its deagglomeration and dispersion into aqueous solutions. As well, formulation efforts were started which were concerned with the formation of stable aqueous solutions of carbon black nanophase particles. The successful approach and knowledge of analysis techniques gained during the analysis of antimony tin oxide in particular, was again applied in the formulation of new aqueous and non-aqueous phase dispersions of the other nanophase powders. Carbon black nanophase particles were tested to learn more about their properties supporting the dispersion of these materials and further supporting the ESD program development efforts. The more highly conducting

sample (XE2) had a very large surface area leading to high solvent absorption. As such, a well-dispersed solution could not be obtained in appropriate concentrations to support the test. A second carbon black (L) was also prepared for analysis. Dispersions were difficult but a solution of appropriate concentration was obtained in very acidic conditions (pH $\approx$ 2). Tests of the isoelectric point were, however, ineffective due to the particle properties in solution. The only conclusions from these tests were that carbon black dispersions in very acidic solutions were possible.

Isoelectric point determinations were not carried out on the fluorine doped ZnO sample due to the small amount available. However, since the isoelectric point of ZnO (pH = 9)[1] is well established, that data has been used as a guide to a good pH range for obtaining good dispersions. In this case, any pH away from the pH of 9 should be appropriate for good dispersion. This observation has some bearing in the addition of ZnO to the melamine formaldehyde resin as the pH of the resin is in the range of 9 – 10.

Several methods of nanophase dispersion had been evaluated during the previous reporting period including combinations and permutations of high shear dispersion methods and ultrasonic techniques, particularly with respect to antimony tin oxide dispersions. In this reporting period, METSS also examined ball milling, another method of effecting dispersion and deagglomeration, using a planetary mill. Solutions of nanophase particles could be put into the ball mill and left to be ground up with the possibility of obtaining deagglomerated nanophase particles. METSS evaluated a 5 wt% and two 45 wt% solutions of $SbSnO_2$ in water using a planetary ball mill. The milling technique proved to make a difference to particle size only if the particle concentration in the solution was large. If it was too small, the effect of the grinding process in the mill was negative (i.e., the technique caused agglomeration). It was observed that different grinding media had different success rates. Comparative observations of the solutions before and after grinding indicated that the viscosity of that solution had increased significantly suggesting that the mill had been effective at breaking up the particles. Particle size analyses were not run immediately after grinding due to unavailability of the equipment. The average particle size determinations were run three days later on a solution with particles that had agglomerated and begun to precipitate out of solution and the results indicated a large average particle size (>500nm). The fact that the particles began to agglomerate and precipitate out of solution after sitting still for several days is of concern however, the same results are not obtained with solutions of the same nanophase particle but at a much lower concentration. If a milling technique were to be used, the resulting solution would have to be diluted immediately after milling to decrease the chances of reagglomeration.

Having established procedures for the use of each technique from the studies with antimony tin oxide (results shown in the July/August bimonthly report), METSS applied those same procedures to other nanophase additives. Using information from the isoelectric point determination test results, METSS carried out a series of particle deagglomeration experiments on a carbon black sample at pH = 2, using the sonic bath and the sonic horn. The dispersion was unstable in the particle analyzer, not allowing particle size estimates to be made. In addition, the carbon black dispersions were unstable over a 24 hour time period. To improve on the overall stability, METSS incorporated polyvinyl alcohol into the aqueous suspension and obtained the desired dispersion.

Fluorine doped ZnO has been examined as a nanophase additive. The sample was received as a resin-compatibilized course powder (compatibilized with a carboxyl functional polyester oligomer). Initial attempts to disperse the powder in deionized, distilled water were unsuccessful, as the powder could not be wetted by the water regardless of the physical methods of dispersion that were tried. A particle-wetted solution was obtained with the addition of a dispersing agent, Duromax D-3021 in 1 wt%, from Rohm and Haas and 2 separate physical methods for dispersion were used. Preliminary particle break up which converted the powder from a coarse powder to a fine powder (average particle size of 600 nm) was

performed using an Ultra Turrex high shear mixer. Although the particle size had decreased, the dispersion was still not stable. As with the antimony tin oxide, high shear methods were unsuccessful for deagglomeration of zinc oxide. However, unlike the studies with antimony tin oxide, zinc oxide could not be sonicated using the sonic horn as the high energy bombardment of the nanophase powder resulted in decomposition (a very apparent burning smell was generated after less than 10 minutes of sonicating using the sonic horn and the formation of black particulate material in the solution was observed). These results necessitated another approach and since it was already known that the ZnO samples could be wetted using Duromax D-3021, this solution was used as the starting point for further investigations into stabilizing colloids. Several samples were prepared using the ZnO/Duromax aqueous solution mixed with a variety of readily available dispersants and each test was done in high and low pH solutions. The results are shown in Table 1. When the dispersion problem was solved, a foaming problem became evident. As a result, several small samples were made using the successful formulation described in Table 1 and three different defoamers were added, a commercial defoamer from Rohm and Haas, isopropyl alcohol and acetone. The only chemical that could be added without destabilizing the dispersion was acetone and it has been employed in the formulation since that time.

Table 1. ZnO Dispersion Stabilization Study.

Stabilizer	Amount	Observations at high pH	Observations at low pH
Acrysol WS-24	1 - 2 wt. %	Unstable dispersion	Unstable dispersion
Acrysol WS-68	1 – 2 wt. %	Unstable dispersion	Unstable dispersion
Acrysol I-62	1 – 2 wt. %	Unstable dispersion	Unstable dispersion
Tamol 165A	1 – 2 wt. %	Unstable dispersion	Unstable dispersion
Acrysol WS-32	1 - 2 wt. %	Unstable dispersion	Stable dispersion$^{\infty}$

* All starting solutions were composed of 5wt% ZnO, 1wt% Duramax D-3021, and 94 wt% water.

$^{\infty}$ Addition of 5 wt.% Duramax D-3021 was necessary to achieve stability.

A second approach to ZnO dispersion included attempts to disperse the ZnO(1%F) powder at 1 wt% in the organic (and melamine compatible) solvent, isopropylalcohol. In this case, the powder was successfully wetted, but a stable dispersion was not obtained. Addition of 0.5 wt% stearic acid, as a dispersing agent proved unsuccessful, even after and hour of sonicating in the sonic bath. Similarly, additions of Duromax D-3021 did not stabilize the ZnO particles in solution.

Carbon fibers were incorporated into laminates in order to test their ability to act as electrostatic dissipating laminate additives. A dispersion of carbon fibers (5 wt%) in water was accomplished with 30 minutes of sonication in the sonic bath. After this amount of time, the fibers were uniformly dispersed and remained so for as long as they were kept before use (up to a week). Similarly, a dispersion of the carbon fibers in isopropyl alcohol could be made with a lower energy high shear method using the Ultra Turrex high shear mixer. In the same manner, the dispersion remained stable for many days after initial dispersion. Both these solutions were added successfully to the melamine formaldehyde resin used to impregnate the cellulose paper. The paper was dried and then formed into a laminate along with the appropriate substrate papers. No attempt to measure the average particle size was made since the fibers are not spherical and the software for the calculations in data analysis for the particle analyzer is based on this assumption.

A series of laminates were prepared from substrate materials and cellulose papers impregnated with solutions of varying ratios of Baytron P to melamine formaldehyde resin. The Baytron P was obtained as a 1.3% solution in water and was easily incorporated into the aqueous melamine formaldehyde resin with high shear mixing to ensure that the two solutions were properly mixed.

Irgastat P22 is initially obtained as a yellow pellet type material. Having just received the material, METSS will be conducting the appropriate formulation tests to try to incorporate it into a laminate. At present, it is known that the product is a high melting (T_{mpt} = 220°C), extensively cross-linked product that does not have any solubility properties in any known solvent.

Table 2 gives an overview of the preliminary laminate formulations.

Table 2. Formulations of Preliminary Laminates

Nanophase Material	pH	Weight % Particle	Weight % Resin	Volume % Particle	Volume % Resin	Final Appearance
$SbSnO_2$	2.8	5	95	0.6	99.4	Hazy blue
$SbSnO_2$	11.6	5	95	0.06	99.4	Hazy blue
ZnO(1%F)$^{\infty}$	6.2	2	98	0.2	99.8	Clear and colorless
ZnO(2%F)$^{\infty}$	7.8	2	98	0.2	99.8	Clear and colorless
C black (L)	0	0.2	99.8	0.6	99.4	Black/Grey
C black (L)	0	0.08	99.92	0.16	99.84	Hazy Grey
C black (L)*	2	0.08	99.92	0.16	99.84	Black/Grey
C black (L6)		0.18	99.82	0.6	99.4	Black/Grey
C fibers	2	1.6	98.4	0.6	99.4	Speckled Black
Baytron	2-3	5	95			Light Blue
Baytron$^{\delta}$	2-3	0.3	99.7			Transparent Very light blue
Baytron$^{\delta}$	2-3	1.0	99.0			Slightly Transparent Streaked blue

• The melamine formaldehyde resin density is ρ = 1.215-1.235 g/cm^3 depending on the batch., $\rho(SbSnO_2)$ = 6.43g/cm^3, ρ(CB L) = 0.400g/cm^3, ρ(CB L6) = 0.350g/cm^3, ρ(C fibres) = 2.0g/cm^3, $\rho(ZnO_2)$ = 5.6g/cm^3

* Surfynol surfactant in ethylene glycol was added at 0.5 wt % of total mixture weight.

∞ 5 wt% Duromax D-3021 and 2 wt% Tamol were added to a pH regulated solution of ZnO to obtain a stable dispersion.

δ Samples prepared at Formica in Cincinnati.

In all of these experiments, the issue of volume percent of an additive vs. weight percent was addressed. Carbon black is a much lighter additive than the other powders that METSS examined and the resin could not stabilize as large a weight fraction in this case.

Initial ESD Test Results

METSS has prepared a series of laminates consisting of a blend of nanophase additives or a commercially available intrinsically conductive polymer (ICP) with the standard melamine formaldehyde resin used by Formica in the production of their commercial laminates. Solution concentrations up to 10% of additive or ICP have been formulated. Test results in the case of the ICPs indicate that less than 1% ICP content may be adequate to address the ESD requirements set forth by the Air Force. In the majority of cases, the acidic pH of the additives in the aqueous dispersion has allowed METSS to replace the current curing agent (*p*-toluenesulfonic acid referred to as Part B) used by Formica to cure their melamine resins with a new Part B that contains the acidic ICP additive or nanophase dispersion.

The laminates were tested using a charge plate monitor IAW standard Air Force practices to determine their effectiveness at dissipating static charge. Test measurements were also performed on three control samples provided by the Air Force at the same time as the METSS sample measurements. All samples were conditioned at the test conditions at least 24 hours prior to ESD measurements. Testing was performed at room temperature down to 20% RH (the extent of our laboratory capabilities).

The ESD performance of the METSS ICP-B formulation was evaluated using a series of laminates prepared by METSS under the same materials and processing conditions used by Formica in the production of their commercial laminates. The only difference between the METSS laminates and the commercial products was that the top sheet impregnated with a solution containing varying concentrations of the METSS ICP B formulation. Each sheet was dried prior to placement on top of a conventional laminate structure (un-cured) and the system was cured at 270°F and 1500 psi. Table 3 shows results of the ESD charge plate monitor tests for ICP B samples tested at different relative humidities and produced by METSS during this last reporting period.

Table 3: ESD Test Results: ICP B Samples measured at 23°C.

Sample Designation	ESD Results (Standard deviation) 40% RH	30%RH	20%RH
1% B	126.7 (3.9)	32.8 (0.3)	167.8 (10.4)
1% C	112.0 (7.8)	57.2 (4.5)	22.2 (6.2)
1% D	14.8 (1.3)	32.8 (0.6)	11.2 (0.3)
5% A	25.5 (2.3)	31.5 (0.3)	17.0 (1.0)
5% B	61.5 (2.3)	31.5 (0.3)	16.0 (0.9)
5% C	71.8 (6.6)	84.5 (6.6)	15.8 (1.4)
5% D	22.8 (5.6)	31(0)	13.5 (0.3)
7% A	20.5 (2.9)	31.2 (0.3)	15.8 (2.4)
7% B	20.0 (4.4)	145.2 (12.6)	23.0 (3.8)
7% C	12.8 (0.3)	40.8 (6.0)	82.2 (7.2)
7% D	23.2 (6.3)	36.2 (3.1)	21.8 (1.1)
10% A	17.5 (2.0)	47.8 (6.2)	21.2 (4.0)
10% B	37.8 (6.4)	115.2 (7.8)	17.2 (1.9)

METSS ICP-B clearly passes the Air Force requirement for less than 200V of remaining charge after 5 seconds of dissipation from 1000V at all humidity levels tested at loading levels down to the 1% tested. Based on the trends in the data, this sample should pass 10%RH testing and loading levels may be able to be reduced to less than 1% active concentration. Due to the nature of the additive, the physical properties of the melamine laminate should not be compromised. The only apparent limitation of this particular formulation is that it is currently opaque with a light colored tint. However, discussions with Formica have indicated that this should not be a hindrance to product commercialization efforts due to the technical nature of the product. The commercial availability of the ICP used in the METSS ICP-B formulations and the ability of METSS to support the *direct replacement* initiative should ensure rapid technology transfer and product commercialization. Formica is very interested in supporting ESD commercialization efforts and is committed to working with METSS to effect technology transfer and scale-up under a very applied Phase II program. METSS was very pleased with the ESD charge plate monitor test results on the 6x6-square inch samples. They were well below the Air Force criteria of less than 200V charge left on the plate and the samples have been sent to the Air Force for further testing.

It was well noted by the Air Force and in the literature that the most significant problem with previously developed laminates was their inability to dissipate charge at lower humidities. In the majority of these cases, the laminate ESD properties were developed using humectants (salts, especially NH_4^+ salts) as additives. The mechanism of charge dissipation relied on humidity and it is not surprising that they were not acceptable performers at lower humidities. METSS has focused its research and development on additives which should not require the presence of water in order to effectively dissipate the charge from

a surface. METSS was very encouraged by the lack of dependence of the charge plate monitor results for our ICP B formulated samples on humidity.

The formulation development efforts associated with the use of nanophase additives have been hindered by the difficulty of developing finely dispersed and stable aqueous phase solutions based on these materials. While we have had a couple of nanophase formulations demonstrate ESD capabilities in the low 200V range, we have yet to develop a stable formulation that can meet the sub-200V ESD requirement set forth by the Air Force. METSS will continue to address this challenge in an effort to ensure an alternative solution is available to support the Phase II efforts. METSS has made tremendous strides working with various nanophase additives systems and still believes this approach can be successfully implemented to meet the program objectives. Results of in-house ESD testing are shown in Tables 4.

Table 4: ESD Test Results. Antimony Tin Oxide samples measured at 23°C.

Replicate ID	Active Concentration	Ave. ESD Results (Standard Deviation)		
		40% RH	30% RH	20% RH
Antimony Tin Oxide				
A	5%	508.0 (71.2)	560.3 (29.0)	636.8 (12.6)
B	5%	632.3 (15.5)	581.8 (13.9)	804.3 (10.9)
A	5%	311.8 (35.1)	192.3 (32.8)	502.3 (65.6)
C	5%	403.8 (9.6)	278.5 (9.5)	463.8 (15.2)
D	5%	331.0 (25.3)	412.3 (24.4)	630.0 (38.1)
ZnO (1%F)				
1	2%	223.0 (4.6)		
2	2%	844.4 (16.7)		
3	2%	511.4 (11.2)		
Carbon Black				
2a	0.08%	460 (111)		
2b	0.08%	748 (160)		
Carbon Fibers				
CF 001	5% A	458.2 (5.5)	607.5 (12.6)	632.0 (27.8)
CF 001	5% B	434.5 (11.6)	648.0 (12.0)	670.2 (9.0)
CF 001	5% C	446.0 (12.2)	602.8 (15.0)	691.5 (6.9)
CF 002	4% A	354.5 (12.7)	613.5 (4.9)	697.0 (7.6)
CF 002	4% B	472.2 (10.8)	558.5 (6.4)	675.2 (10.4)
CF 002	4% C	400.5 (2.1)	614.5 (8.3)	657.8 (12.7)
CF 003	3% A	431.0 (11.90	172.2 (2.8)	583.2 (1.9)
CF 003	3% B	557.0 (15.4)	580.8 (6.2)	687.8 (11.8)
CF 003	3% C	350.7 (6.7)	579.8 (18.6)	739.0 (7.0)
CF 004	2% A	400.2 (11.9)	181.5 (3.6)	469.2 (6.6)
CF 004	2% B	341.5 (5.8)	613.5 (3.8)	667.8 (6.0)
CF 004	2% C	393.7 (14.3)	505.8 (9.7)	578.5 (9.9)
CF 005	1% A	428.2 (10.8)	697.5 (13.9)	710.5 (5.6)
CF 005	1% B	463.2 (12.1)	546.5 (7.9)	741.5 (10.0)

Production Runs At Formica

METSS has performed two lab-scale production runs at Formica in Cincinnati, Ohio to produce the 18x18-inch samples required by the Air Force for testing. The production runs went very well and clearly demonstrated the *direct replacement* aspect of the technology METSS was striving to achieve. The only thing that was different between their process for non-ESD laminates and the runs performed by Formica and METSS to support the SBIR program was the part B material that was used - they used our Part B curative (with ESD additive) and processed the ESD laminates in the exact same way as the conventional laminates. They were impressed by how well our material went on and cured, as well as the look and quality of the final product.

During the first visit we ended up making more material than we had planned and as a consequence did not have enough of our Part B to make the targeted 1% additive concentration in the final product (which we know will pass based on in-house ESD test results, see Table 3). We ended up with an active concentration of 0.3% when processing the laminates to meet the 64% resin content on the cellulose top sheet typically targeted by Formica. When tested, these samples fully dissipate the 1000V charge when the probe is in contact but the charge on the plate goes back up when it is removed. However, these samples clearly demonstrated the direct replacement aspect of the METSS technology and our ability to produce high quality and aesthetically pleasing laminates.

A second set of samples was prepared during a second visit to Formica using enough of the Part B ESD additive to make an ESD laminate at 1% active concentration. The appearance of these samples was much darker than the 0.3% samples and processing difficulties were observed that led to an uneven distribution of resin (and hence METSS ICP-B additive). These difficulties were related to the higher dilution factor of the resin system caused by the addition of proportionally more of a very dilute Part B additive (1.3% by weight). In the second case only 54% resin was added to the cellulose top layer during processing due to the high water content of the resin with the additive. Thus, the target of 1% additive in the cellulose top sheet was still not met. High concentration areas viewed as streaks and spots are noticeable on the final laminates and the ESD properties are not optimized. ESD samples of the tests indicated that the samples were able to fully dissipate the 1000V charge when the probe is in contact but the charge on the plate goes back up to about 700V once it is removed. One sample was recoated, after drying, using a post processing technique to increase the amount of resin added to the laminate to the target amount of 64%. ESD tests indicated that the laminate could dissipate the 1000V charge completely in the time required. The results are shown in Table 5.

METSS has included samples for which ESD testing was done by the Air Force. The first four samples are 6" x 6" laminates, two with 1% ICP and two with 5% ICP. They were prepared by METSS at our facility and clearly show the technical feasibility of the ICP system, through their ESD test results. The next four samples, are 0.3% ICP samples prepared at Formica in our first attempt at larger scale laminate production. Although these samples do not pass the dissipation criteria set forth by the Air Force, they do indicate that METSS ICP Part B can be incorporated successfully into the existing process at Formica as the resin-curing catalyst to give aesthetically pleasing laminates. The last six samples, also made at Formica, contain 1% ICP B in the resin. Due to processing difficulties, these samples do not exhibit the superior laminate appearance of the less concentrated samples. These production difficulties have resulted in unevenly coated papers with areas of higher and lower ICP B concentration. As well, the laminate itself does not have the usual amount of resin added in it. In most instances, Formica aims for 64% resin on the cellulose sheet. In the case of five of this last group of laminates, only 54% resin was added due to the high water content of the resin with the additive. The last laminate, ICP B 014 was modified to effectively increase the amount of resin on the laminate to nearer 64%. The importance of the proper ratio of resin to paper is shown in the

data. The laminate with the target amount of resin has superior ESD characteristics when compared to the laminate with only 54% resin content.

Table 5. AFRL Test Results

Sample Descriptor	Sample Size	METSS ICP-B	METSS ESD Results (RT, 30%RH)	Surface Resistance	AFRL Testing Pt. to Pt. Resistance	AF ESD Results
ICP B 001	6" x 6"	1%	127 ± 4	5.9×10^6	2×10^7	22 V
ICP B 002	6" x 6"	1%	15 ± 1	3.4×10^6	1.9×10^7	33 V
ICP B 003	6" x 6"	5%	26 ± 2	1.14×10^6	1.1×10^6	
ICP B 004	6" x 6"	5%	61 ± 2	*4.2×10^5	5.7×10^5	
ICP B 005	18" x 18"	0.3%	>200	7.8×10^9	5.6×10^7	
ICP B 006	18" x 18"	0.3%	>200	6.3×10^9	6.9×10^9	
ICP B 007	18" x 18"	0.3%	>200	4.2×10^9	5.3×10^9	
ICP B 008	18" x 18"	0.3%	>200	3.7×10^{10}	9.3×10^9	
ICP B 009	18" x 18"	<1%	200 - 400	1.4×10^8	1.2×10^7	
ICP B 010	18" x 18"	<1%	>200	3.1×10^7	1.5×10^7	599 (+) 220 (-)
ICP B 011	18" x 18"	<1%	100-300	2.5×10^7	1.4×10^7	387 (+) 38 (-)
ICP B 012	18" x 18"	<1%	>200	2.6×10^7	2.3×10^7	
ICP B 013	18" x 18"	<1%	>200	1.6×10^7	1.7×10^7	
ICP B 014	18" x 18"	1%	<100	5.4×10^7	*8.7×10^5	<15 V

*tested at 10V

Anticipated Progress (Next Reporting Period)

- METSS will continue work to investigate the potential of nanophase additive technologies.
- METSS will continue to communicate with Formica and advance the project through interactions with them and joint production of samples.
- METSS will continue to communicate with the Air Force and utilize their expertise by sending qualifying samples further testing.
- METSS will continue to accumulate and evaluate data to further advance the project.

Phase I Work Schedule
Projected vs. Actual Performance

Task Description	Month								
	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
Task 1. Start of Work Meeting	◆								
Progress	◆								
Task 2. Technical Review									
Progress									
Task 3. Processing & Fabrication Development									
Progress									
Task 4. Testing and Evaluation									
Progress									
Task 5. Prototype Fabrication and Testing									
Progress									
Task 6. Administrative & Reporting			M		M		M		M F

M = Monthly Progress Report; F = Final Report

METSS CONFIDENTIAL

7 10

METSS

Memo

To: Julius Brodbeck

CC: Mike Manders, Steve Gerken, Michael Hitchcock

From: K. Heater

Date:

Re: Phase I Technical Feasibility Demonstration

The program proposal submitted by METSS emphasized two approaches to address the production of hard ESD laminate surfaces:

1. use of proper additives (nanophase)
2. polymer blending using intrinsically conducting polymers or co-polymers.

Furthermore, METSS emphasized the development of *direct replacement technologies*, i.e. technologies that can be readily integrated into commercial laminate production processes without changing process equipment or parameters.

A significant amount of work has gone into developing and testing formulations based on both of the proposed approaches and METSS is pleased to report that it has clearly demonstrated the technical feasibility of using one of these approaches to meet the technical objectives of the program and has made significant inroads in developing the other. The results are briefly summarized in this memo.

METSS ICP-B ESD Laminate Formulation

METSS has prepared a series of laminates consisting of a blend of a commercially available intrinsically conductive polymer (ICP) with the standard melamine formaldehyde resin used by Formica in the production of their commercial laminates. These formulations are stable and 100% water soluble (no VOCs). Solution concentrations up to 10% ICP have been formulated, but test results indicate that less than 1% ICP content may be adequate to address the ESD requirements set forth by the Air Force. The chemistry of the system should essentially allow METSS to replace the current curing agent (Part B) used by Formica to cure their melamine resins with a new Part B that contains the ICP additive. The METSS ICP-B is an aqueous solution that mixes readily with the melamine resin system and has the right pH to effect cure in the same manner as the existing Part B curative. Thus, with the exception of switching to a new Part B curative (METSS ICP-B), production methods for the ESD laminate systems should be unchanged.

The ESD performance of the METSS ICP-B formulation was evaluated using a series of laminates prepared by METSS under the same materials and processing conditions used by Formica in the production of their commercial laminates. The only difference between the METSS laminates and the commercial products was that the top sheet impregnated with a solution containing varying concentrations of the METSS ICP B formulation. Each sheet was dried prior to placement on top of a conventional laminate structure (un-cured) and the system was cured at 270°F and 1500 psi.

The laminates were tested using a charge plate monitor IAW standard Air Force practices to determine their effectiveness at dissipating static charge. Test measurements were also performed on three control samples provided by the Air Force at the same time as the METSS ICP-B measurements. All samples were conditioned at the test conditions at least 24 hours prior to ESD measurements. Testing was performed at room temperature down to 20% RH (the extent of our laboratory capabilities). The results for laminates produced using a 10wt%, a 5wt%, and a 1wt% solution of conductive additive in the ICP B are provided in Figure 1. An expanded plot is provided in Figure 2.

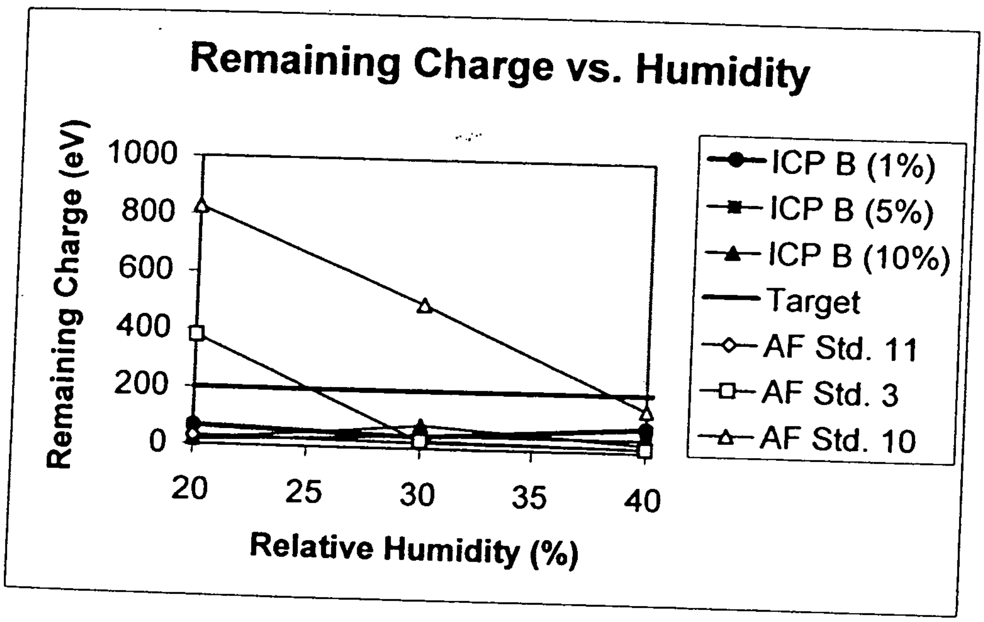

Figure 1. ESD Test Results - METSS ICP-B

METSS ICP-B clearly passes the Air Force requirement for less than 200eV of remaining charge after 5 seconds of dissipation from 1000eV at all humidity levels tested at loading levels down to the 1% tested. Based on the trends in the data, this sample should pass 10%RH testing and loading levels may be able to be reduced to less than 1% active concentration. Due to the nature of the additive, the physical properties of the melamine laminate should not be compromised. The only apparent limitation of this particular formulation is that it is currently opaque with a light colored tint. However, discussions with Formica have indicated that this should not be a hindrance to product commercialization efforts due to the technical nature of the product. The commercial availability of the ICP used in the METSS ICP-B formulations and the ability of METSS to support the *direct replacement* initiative should ensure rapid technology transfer and product commercialization. Formica is very interested in supporting ESD commercialization efforts and is committed to working with METSS to effect technology transfer and scale-up under a very applied Phase II program.

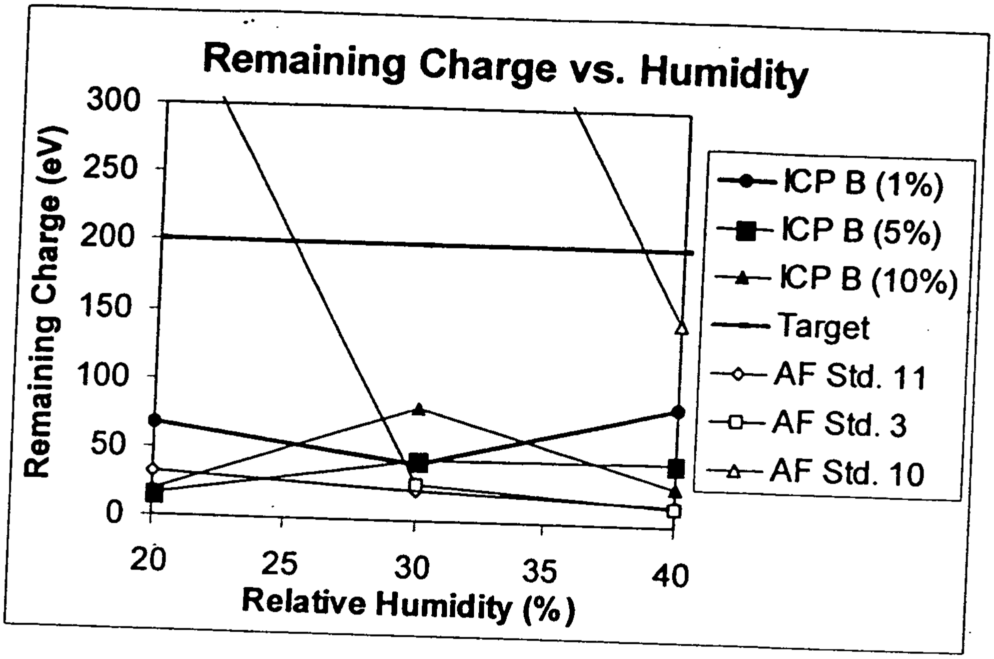

Figure 2. Figure 1, expanded.

Ongoing Efforts

Development efforts are continuing with other ICP systems (total of 4 candidates) that METSS has obtained to support the ESD formulation development efforts. In addition, METSS has made tremendous strides working with various nanophase additives systems and still believes this approach can be successfully implemented to meet the program objectives. However, the formulation development efforts associated with the use of nanophase additives have been hindered by the difficulty of developing finely dispersed and stable aqueous phase solutions based on these materials. While we have had a couple of nanophase formulations demonstrate ESD capabilities in the low 200eV range, we have yet to develop a stable formulation that can meet the sub-200eV ESD requirement set forth by the Air Force. METSS will continue to address this challenge in an effort to ensure an alternative solution is available to support the Phase II efforts.

Production Runs At Formica

Since the original memo was drafted, METSS has performed two lab-scale production runs at Formica in Cincinnati, Ohio to produce the 18x18-inch samples required by the Air Force for testing. The production runs went very well and clearly demonstrated the *direct replacement* aspect of the technology METSS was striving to achieve. The only thing that was different between their process for non-ESD laminates and the runs performed by Formica and METSS to support the SBIR program was the part B material that was used - they used our Part B curative (with ESD additive) and processed the ESD laminates in the exact same way as the conventional laminates. They were impressed by how well our material went on and cured, as well as the look and quality of the final product.

First Production Run at Formica

During the first visit was that we ended up making more material than we had planned and as a consequence did not have enough of our Part B to make the targeted 1% additive concentration in the final product (which we know will pass based on in-house ESD test results). We ended up with an active concentration of 0.3% when processing the laminates to meet the 64% resin content on the cellulose top sheet typically targeted by Formica. When tested, these samples fully dissipate the 1000eV charge when the probe is in contact but the charge on the plate goes back up when it is removed. However, these samples clearly demonstrated the direct replacement aspect of the METSS technology and our ability to produce high quality and aesthetically pleasing laminates.

Second Production Run at Formica

A second set of samples was prepared during a second visit to Formica using enough of the Part B ESD additive to make an ESD laminate at 1% active concentration. The appearance of these samples was much darker than the 0.3% samples and processing difficulties were observed that led to an uneven distribution of resin (and hence METSS ICP-B additive). These difficulties were related to the higher dilution factor of the resin system caused by the addition of proportionally more of a very dilute Part B additive (1.3% by weight). In the second case only 54% resin was added to the cellulose top layer during processing due to the high water content of the resin with the additive. Thus, the target of 1% additive in the cellulose top sheet was still not met. High concentration areas viewed as streaks and spots are noticeable on the final laminates and the ESD properties are not optimized.

Summary

Our current results suggest that this system can be further optimized to address the processing issues and to provide the proper balance between performance and aesthetics. Aside from varying the concentration of the additive in the METSS ICP-B Part B formulation to achieve the desired resin content in the top layer of the laminate, the formulation may be modified through the addition of other conductive additive systems, either conventional or nanophase, to achieve the proper balance of properties. Based on the present results, METSS is extremely confident we will be able to meet this objective.

The commercialization path through Formica looks very good. Even the economics of the proposed technology should be satisfactory. Formica is very excited about pursuing this effort further and will be working closely with METSS under the Phase II program.

Description of ESD Samples

Rt to Pt

100V 15sec Conc...

Sample Descriptor	Sample Size	% METSS ICP-B	ESD Results (RT 30% RH)	~~Conductivity~~ Resistance surface	AF ESD Results	surface resistance Ω
METSS ICP B 001	6" x 6"	1%	127 ± 4	2x10^7	22	5.9x10^6
METSS ICP B 002	6" x 6"	1%	15 ± 1	1.9x10^7	33	3.4x10^6
METSS ICP B 003	6" x 6"	5%	26 ± 2	1.1x10^6		1.14x10^6
METSS ICP B 004	6" x 6"	5%	10V 61 ± 2	5.75x10^5		4.2x10^5 10V
METSS ICP B 005	18" x 18"	0.3%	980 ± 14	5.6x10^9		7.8x10^9
METSS ICP B 006	18" x 18"	0.3%	972 ± 10	6.9x10^9		6.3x10^9
METSS ICP B 007	18" x 18"	0.3%	965 ± 22	5.3x10^9		4.2x10^9
METSS ICP B 008	18" x 18"	0.3%	982 ± 4	9.3x10^9		3.6x10^10
METSS ICP B 009	18" x 18"	1%	317 ± 119	1.19x10^7		1.4x10^8
METSS ICP B 010 Rough edge	18" x 18"	1%	490 ±148	1.47x10^7	599/220	3.1x10^7
METSS ICP B 011	18" x 18"	1%	225 ± 107	1.9x10^7	387/38	2.5x10^7
METSS ICP B 012	18" x 18"	1%	477 ± 175	2.3x10^7		2.6x10^7
METSS ICP B 013	18" x 18"	1%	582 ± 140	1.7x10^7		1.6x10^7

METSS has included samples for ESD testing by the Air Force. The first four samples are 6" x 6" laminates, two with 1% ICP and two with 5% ICP. They were prepared by METSS at our facility and clearly show the technical feasibility of the ICP system, through their ESD test results. The next four samples, are 0.3% ICP samples prepared at Formica in our first attempt at larger scale laminate production. Although these samples do not pass the dissipation criteria set forth by the Air Force, they do indicate that METSS ICP Part B can be incorporated successfully into the existing process at Formica as the resin-curing catalyst to give aesthetically pleasing laminates. The last five samples, also made at Formica, contain 1% ICP B in the resin. Due to processing difficulties, these samples do not exhibit the superior laminate appearance of the less concentrated samples. These production difficulties have resulted in unevenly coated papers with areas of higher and lower ICP B concentration. As well, the laminate itself does not have the usual amount of resin added in it. In most instances, Formica aims for 64% resin on the cellulose sheet. In the case of this last group of laminates, only 54% resin was added due to the high water content of the resin with the additive.

9 H2 2x10^7 R140pt 9.7x10^6

blue edge